

EXHIBIT B

REPORTING AND DELIVERABLES REQUIREMENTS

## Exhibit B - Reporting and Deliverables Requirements

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Exhibit B--Section 1  
Contract Reports/Deliverables Distribution

1.0 CONTRACT REPORTS/DELIVERABLES DISTRIBUTION

- 1.1 35-Day Data Turnaround. The following table specifies the contract reporting and deliverable requirements that must be distributed to the Regional Sample Control Center (RSCC).

NOTE: Specific recipient names and addresses are subject to change during the term of the contract. The EPA shall notify the Contractor in writing of such changes when they occur.

Table 1

	Item	No. of Copies	Delivery Schedule	RSCC
A. <sup>1</sup>	Sample Traffic Reports	1	5 working days after receipt of last sample in Sample Delivery Group (SDG). <sup>2</sup>	X
B. <sup>3,4</sup>	Complete SDG File	1	35 days after receipt of last sample in SDG	X
C. <sup>1</sup>	Annual Verification of Method Detection Limits	1	Annually: 15th day of January.	X
D. <sup>3</sup>	Results of Quarterly Blind PE Sample	2	35 days after VTSR of last sample in SDG.	X
E. <sup>5</sup>	Standard Operating Procedures	1	28 days after contract award, and as required in Exhibit E.	As directed
F. <sup>5</sup>	Laboratory Quality Assurance Plan	1	28 days after contract award, and as required in Exhibit E.	As directed
G.	GC/MS Tapes	Lot	Retain for 365 days after data submission  Submit within 7 days after receipt of written request by EPA	As directed
H.	GC Tapes	Lot	Retain for 365 days after data submission  Submit within 7 days after receipt of written request by EPA	As directed
I.	Extracts	Lot	Retain for 365 days after data submission  Submit within 7 days after receipt of written request by EPA	As directed

Footnotes:

- 1 Also required in the Sample Data Summary Package.
- 2 A sample delivery group (SDG) is a group of samples within a Case, received over a period of 14 days or less (7 days or less for 14-day data turnaround contracts) and not exceeding 20 samples. Data for all samples in the SDG are due concurrently. The date of delivery of the SDG or any samples within the SDG is the date that the last sample in the SDG is received. (See Exhibit A for further description.)
- 3 **DELIVERABLES ARE TO BE REPORTED TOTAL AND COMPLETE. Concurrent delivery required. Delivery shall be made such that the designated recipient receives all items on the same calendar day. This includes resubmissions of any required deliverables. The date of delivery of the SDG, or any sample within the SDG, is the date all samples have been delivered. If the deliverables are due on a Saturday, Sunday or Federal holiday, then they shall be delivered on the next business day. Deliverables delivered after this time will be considered late.**
- 4 Complete SDG File shall contain the original sample data package described in Section 2.6, plus all of the original documents described under Section 2.7.
- 5 See Exhibit E and Exhibit F for a more detailed description.

NOTE: Unless otherwise instructed by the EPA, the Contractor shall dispose of unused sample volume and used sample bottles/containers no earlier than sixty (60) days following submission of the reconciled Complete SDG File. Sample disposal and disposal of unused sample bottles/containers is the responsibility of the Contractor and must be done in accordance with all applicable laws and regulations governing disposal of such materials.

Exhibit B--Section 1  
Contract Reports/Deliverables Distribution

- 1.2 14-Day Data Turnaround. The following table specifies the contract reporting and deliverable requirements that must be distributed to the Regional Sample Control Center (RSCC).

NOTE: Specific recipient names and addresses are subject to change during the term of the contract. The EPA will notify the Contractor in writing of such changes when they occur.

Table 2

	Item	No. of Copies	Delivery Schedule	RSCC
A. <sup>1</sup>	Sample Traffic Reports	1	5 working days after receipt of last sample in Sample Delivery Group (SDG). <sup>2</sup>	X
B. <sup>3,4</sup>	Complete SDG File	1	14 days after receipt of last sample in SDG	X
C. <sup>1</sup>	Annual Verification of Method Detection Limits	1	Annually: 15th day of January.	X
D. <sup>3</sup>	Results of Quarterly Blind PE Sample	2	14 days after receipt of last sample in SDG.	X
E. <sup>5</sup>	Standard Operating Procedures	1	28 days after contract award, and as required in Exhibit E.	As directed
F. <sup>5</sup>	Laboratory Quality Assurance Plan	1	28 days after contract award, and as required in Exhibit E.	As directed
G.	GC/MS Tapes	Lot	Retain for 365 days after data submission  Submit within 7 days after receipt of written request by EPA	As directed
H.	GC Tapes	Lot	Retain for 365 days after data submission  Submit within 7 days after receipt of written request by EPA	As directed
I.	Extracts	Lot	Retain for 365 days after data submission  Submit within 7 days after receipt of written request by EPA	As directed

Footnotes:

- 1 Also required in the Sample Data Summary Package.
- 2 A sample delivery group (SDG) is a group of samples within a Case, received over a period of 14 days or less (7 days or less for 14-day data turnaround contracts) and not exceeding 20 samples. Data for all samples in the SDG are due concurrently. The date of delivery of the SDG or any samples within the SDG is the date that the last sample in the SDG is received. (See Exhibit A for further description.)
- 3 **DELIVERABLES ARE TO BE REPORTED TOTAL AND COMPLETE. Concurrent delivery required. Delivery shall be made such that the designated recipient receives all items on the same calendar day. This includes resubmissions of any required deliverables. The date of delivery of the SDG, or any sample within the SDG, is the date all samples have been delivered. If the deliverables are due on a Saturday, Sunday or Federal holiday, then they shall be delivered on the next business day. Deliverables delivered after this time will be considered late.**
- 4 Complete SDG File shall contain the original sample data package described in Section 2.6, plus all of the original documents described under Section 2.7.
- 5 See Exhibit E and Exhibit F for a more detailed description.

NOTE: Unless otherwise instructed by the EPA, the Contractor shall dispose of unused sample volume and used sample bottles/containers no earlier than sixty (60) days following submission of the reconciled Complete SDG File. Sample disposal and disposal of unused sample bottles/containers is the responsibility of the Contractor and must be done in accordance with all applicable laws and regulations governing disposal of such materials.

Exhibit B--Section 1  
Contract Reports/Deliverables Distribution

- 1.3 Labor Hour Pool (LHP) Data. Sample preparation/analyses which do not conform to the analytical methods described in Exhibit D, shall be analyzed under the Labor Hour Pool (LHP) portion of the contract. Whenever LHP sample preparation/analyses are performed, the Contractor must provide adequate documentation which support all current and future uses of the data. Potential uses of the data can include data validation, monitoring, modelling, risk assessment, site characterization, Record of Decision defense, enforcement, litigation, etc.

Although the nature of the LHP work assignment is undefined, all reports and data deliverables must conform in content to the elements specified in Exhibits B, E and F of the Statement of Work. Tables 1 and 2 specify the contract reports and deliverables that shall be required for LHP analyses and that must be distributed to the Regional Sample Control Center (RSCC). The following is a breakdown of each of the LHP reports and deliverables, along with the appropriate Section reference that contains the requirements that must be addressed:

- 1.3.1 Sample Traffic Reports. Refer to the requirements in Section 2.4.
- 1.3.2 Sample Data Summary Package. Refer to the requirements in Section 2.5. Note: Section 1.3.3 below provides Section references for each of LHP Forms required.
- 1.3.3 Complete SDG File. Complete data packages must contain all of the elements referred to in Sections 2.6 and 2.7, including the SDG Narrative, data reporting Forms, raw data and all records relating to the SDG. Note: Although Sections 2.6.3 through 2.6.10 describe the required elements of the analytical methods in Exhibit D and do not directly relate to LHP analyses, these sections do provide templates that should be used as guidelines for the preparation and organization of LHP GC/MS or GC sample data packages. The following is a summary of the required elements of a Complete SDG File data package:
- 1.3.3.1 GC/MS data package.
- 1.3.3.1.1 SDG Narrative. Refer to Section 2.6.1 for the narrative requirements. In addition to the items required in the narrative, the contractor must attach a copy of the Workplan developed for the LHP analytical method.
- 1.3.3.1.2 Traffic Reports. Refer to Section 2.6.2 for requirements.
- 1.3.3.1.3 LHP data. Although explicit forms have not been developed LHP analyses, the Contractor shall follow the Form style presented in Section 4, the Form descriptions in Section 3 and data presentation format in Section 2 to develop and model their data package. The Contractor shall submit the following LHP Forms along with the relevant raw data:
- ! Form I LHP - Organic Analysis Data Sheet. (Section 3.4)
  - ! Form I LHP-TIC - Organic Analysis Data Sheet: Tentatively Identified Compounds. (Section 3.5)
  - ! Form II LHP - System Monitoring Compound or Surrogate Recovery data sheet. (Section 3.6 and 3.7)
  - ! Form III LHP - Matrix Spike/Matrix Spike Duplicate Recovery data sheet. (Section 3.8)
  - ! Form IV LHP - Method Blank Summary. (Section 3.9)
  - ! Form V LHP - GC/MS Instrument Performance Check and Mass Calibration. (Section 3.10)
  - ! Form VI LHP - GC/MS Initial Calibration Data. (Section 3.11)
  - ! Form VII LHP - Initial Calibration Verification data sheet. (Section 3.15)



- ! Form VIII LHP - Continuing Calibration Data.  
(Section 3.17)
- ! Form IX LHP - Internal Standard Area and Retention Time  
Summary. (Section 3.20)
- 1.3.3.1.4 Records and Remaining Raw Data. Refer to Section 2.7 for the  
remaining requirements that must be submitted with the Complete  
SDG File data package.
- 1.3.3.2 GC data package.
- 1.3.3.2.1 SDG Narrative. Refer to Section 2.6.1 for the narrative  
requirements. In addition to the items required in the  
narrative, the contractor must attach a copy of the Workplan  
developed for the LHP analytical method.
- 1.3.3.2.2 Traffic Reports. Refer to Section 2.6.2 for requirements.
- 1.3.3.2.3 LHP Data. Although explicit forms have not been developed LHP  
analyses, the Contractor shall follow the Form style presented  
in Section 4, the Forms descriptions in Section 3 and data  
presentation format in Section 2 to develop and model their  
data package. The Contractor shall submit the following LHP  
Forms along with the relevant raw data:
  - ! Form I LHP - Organic Analysis Data Sheet. (Section 3.4)
  - ! Form II LHP - Surrogate Recovery data sheet.  
(Section 3.7)
  - ! Form III LHP - Matrix Spike/Matrix Spike Duplicate Recovery  
data sheet. (Section 3.8)
  - ! Form IV LHP - Method Blank Summary. (Section 3.9)
  - ! Form VI LHP - GC Initial Calibration Data. (Section 3.14)
  - ! Form VII LHP - Initial Calibration Verification data sheet.  
(Section 3.16)
  - ! Form VIII LHP - Continuing Calibration Data.  
(Section 3.19)
  - ! Form IX LHP - Internal Standard Area and Retention Time  
Summary. Required if the technique applicable to the LHP  
analytical Method. (Section 3.20)
  - ! Form IX LHP - GC Analytical Summary. (Section 3.21)
  - ! Form X LHP - Clean up Summary. Required if there is a  
sample cleanup procedure employed (e.g., GPC, florisil,  
etc.). (Section 3.22)
  - ! Form XI LHP - Target Compound Identification. Required for  
dual column confirmation GC analyses. (Section 3.23)
- 1.3.3.2.4 Records and Remaining Raw Data. Refer to Section 2.7 for the  
remaining requirements that must be submitted with the data  
package.
- 1.3.4 Standard Operating Procedures. Refer to the requirements in Exhibits  
E and F.
- 1.3.5 Laboratory Quality Assurance Plan. Refer to the requirements in  
Exhibits E and F.
- 1.3.6 GC/MS Tape Audits. Refer to the requirements in Exhibit E, Section  
8.0.
- 1.3.7 GC Tape Audits. Refer to the requirements in Exhibit E, Section 9.0.
- 1.3.8 Extracts. Refer to the requirements in Exhibit B, Section 2.10.

Exhibit B--Section 1  
Contract Reports/Deliverables Distribution

1.3.9 Quarterly Blind Performance Evaluation Program. Refer to the requirements in Exhibit E, Section 7.0.

1.4 Distribution Address.

Region: Regional Sample Control Center (RSCC)  
USEPA New England Regional Laboratory  
60 Westview St.  
Lexington, MA 02421

2.0 REPORTING REQUIREMENTS AND ORDER OF DATA DELIVERABLES

2.1 Introduction. The Contractor shall provide reports and data deliverables of the required content and form as described in this exhibit. All reports and documentation must be:

- Legible,
- Clearly labeled and completed in accordance with instructions in this exhibit,
- Arranged in the order specified in this section,
- Paginated consecutively in ascending order starting from the SDG Narrative, and
- Copies must be legible.

NOTE: Complete SDG files need not be double-sided. (The CSF is composed of original documents.)

2.1.1 Requirements for each deliverable item are specified in Sections 2.3-2.10. Prior to submission, the Contractor shall arrange items and the components of each item in the order listed in these sections.

2.1.2 The Contractor shall use EPA Case numbers (including SDG numbers) and EPA sample numbers to identify samples received under this contract, both verbally and in reports/correspondence. The contract number shall be specified in all correspondence.

2.2 Resubmission of Data. If submitted documentation does not conform to the above criteria, the Contractor shall resubmit such documentation with deficiency(ies) corrected, at no additional cost to the Agency.

2.2.1 The Contractor shall respond within seven (7) days to written requests from data recipients for additional information or explanations that result from the Government's inspection activities unless otherwise specified in the contract.

2.2.2 Whenever the Contractor is required to submit or resubmit data as a result of an on-site laboratory evaluation, or through an EPA action, or through a Regional data reviewer's request, the data shall be clearly marked as ADDITIONAL DATA and shall be sent to the RSCC. The Contractor shall include a cover letter which describes which data are being delivered, to which EPA Case(s) the data pertain, and **who requested the data.**

2.3 Laboratory Quality Assurance Plan and Standard Operating Procedures. The Contractor shall adhere to the requirements in Exhibits E and F.

2.4 Sample Traffic Reports. Each sample received by the Contractor will be labeled with an EPA sample number, and will be accompanied by a Sample Traffic Report (TR) bearing the sample number and descriptive information regarding the sample. The Contractor shall complete the TR (marked for return to the RSCC), recording the date of sample receipt and sample condition upon receipt for each container, and shall sign the TR. Information shall be recorded for each sample in the SDG.

Exhibit B--Section 2  
Reporting Requirements and Order of Data Deliverables

- 2.4.1 The Contractor shall submit TRs in SDG sets (i.e., TRs for all samples in an SDG shall be clipped together), with an SDG cover sheet attached. The SDG cover sheet shall contain the following items:
- Laboratory name,
  - Contract number,
  - Sample analysis price (full sample price from the contract),
  - Case number, and
  - List of EPA sample numbers of all samples in the SDG, identifying the **first** and **last** samples received, and their dates of receipt.
- NOTE: When more than one sample is received in the first or last SDG shipment, the "first" sample received would be the lowest sample number (considering both alpha and numeric designations); the "last" sample received would be the highest sample number (considering both alpha and numeric designations).
- 2.4.2 Each TR shall be clearly marked with the SDG number, entered below the laboratory receipt date on the TR. The TR for the **last** sample received in the SDG shall be clearly marked "SDG-FINAL SAMPLE." The SDG number is the EPA sample number of the first sample received in the SDG. When several samples are received together in the first SDG shipment, the SDG number shall be the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG.
- 2.4.3 If samples are received at the laboratory with multi-sample TRs, all the samples on one multi-sample TR may not necessarily be in the same SDG. In this instance, the Contractor shall make the appropriate number of photocopies of the TR, and submit one copy with each SDG cover sheet.
- 2.5 Sample Data Summary Package. The sample data summary package shall be ordered as follows and shall be submitted separately (i.e., separated by rubber bands, clips or other means) directly **preceding** the complete SDG file. Sample data forms shall be arranged in increasing EPA sample number order, considering **both** letters and numbers. For example, BE400 is a lower sample number than BF100, as E precedes F in the alphabet. The SDG number shall be reported on all data reporting forms. The sample data summary package shall contain data for all samples in one SDG of the Case, as follows. (See Section 2.6 for a detailed description of each item.) The sample data summary package shall be arranged in the same manner as the complete SDG file.
- 2.5.1 SDG Narrative.
- 2.5.2 Arranged by fraction and by sample within each fraction: tabulated target compound results (Form I), including tentatively identified compounds (Form I TIC) for GC/MS methods.
- 2.5.3 Arranged by fraction: system monitoring compound or surrogate spike analysis results (Form II) by matrix (water, soil or waste) and for soil, by concentration (low or medium).
- 2.5.4 Arranged by fraction: matrix spike/matrix spike duplicate results (Form III).
- 2.5.5 Arranged by fraction: method blank data (Form IV), and tabulated blank results (Form I), including tentatively identified compounds (Form I TIC) for GC/MS methods.
- 2.5.6 Arranged by fraction: internal standard area data (Form IX) for volatile, semivolatile, and modified 524.2 fractions only.
- 2.6 Sample Data Package. The sample data package is divided into the ten major units described in this section. The last eight units are each specific to an analytical fraction (volatiles, semivolatiles, pesticides/Aroclors, etc.). If the analysis of a fraction is not

required, then that fraction-specific unit is not required as a deliverable. The sample data package shall include data for the analyses of all samples in one SDG, including field samples, dilutions, reanalyses, blanks, matrix spikes, and matrix spike duplicates. The Contractor shall retain a copy of the sample data package for 365 days after final acceptance of data. After this time, the Contractor may dispose of the package.

- 2.6.1 SDG Narrative. This document shall be clearly labeled "SDG Narrative" and shall contain: laboratory name; Laboratory Code; Case number; EPA sample numbers in the SDG, differentiating between initial analyses and reanalyses; SDG number; Contract number; and detailed documentation of any quality control, sample, shipment and/or analytical problems encountered in processing the samples reported in the data package. All capillary GC columns used for analysis shall be documented here, by fraction. List the column identification/brand name, the length in meters (m), the internal diameter in millimeters (mm), and the film thickness in micrometers ( $\mu\text{m}$ ). The trap used with purge and trap analyses shall be described here. List trap name, when denoted by the manufacturer, its composition (packing material/brand name, amount of packing material, in length, cm). The Contractor shall include any technical and administrative problems encountered, the EPA sample numbers of any affected sample, the corrective actions taken, the resolution, an explanation for all flagged edits (e.g., manual edits) on quantitation lists. The Contractor shall document in the SDG Narrative all instances of manual integration. The SDG Narrative shall contain the following statement, verbatim: "I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package has been authorized by the laboratory manager or his designee, as verified by the following signature." This statement shall be directly followed by an original signature of the laboratory manager or his designee with a typed line below it containing the signer's name and title, and the date of signature.
  - 2.6.1.1 Whenever data from sample reanalyses are submitted, the Contractor shall state in the SDG Narrative for **each** reanalysis whether the reanalysis is billable, and if so, why.
  - 2.6.1.2 The Contractor shall list the pH determined for each water sample submitted for volatiles and modified 524.2 volatiles analyses. This information may appear as a simple list or table in the SDG Narrative. The purpose of this pH determination is to ensure that all water volatiles samples were acidified in the field. No pH adjustment is to be performed by the Contractor on water samples for volatiles analysis.
- 2.6.2 Traffic Reports. The Contractor shall include a copy of the TRs submitted in Section 2.4 for all of the samples in the SDG. The TRs shall be arranged in increasing EPA sample number order, considering both letters and numbers. Copies of the SDG cover sheet are to be included with the copies of the TRs. (See Section 2.4 for more detail on reporting requirements for TRs.) In the case of multi-sample TRs, the Contractor shall make the appropriate number of photocopies of the TR so that a copy is submitted with each applicable data package. In addition, in any instance where samples from more than one multi-sample TR are in the same data package, the Contractor shall submit a copy of the SDG cover sheet with copies of the TRs.
- 2.6.3 Volatiles Data (modified OLM03.1)
  - 2.6.3.1 Volatiles QC Summary
    - 2.6.3.1.1 System Monitoring Compound Summary (Form II VOA).
    - 2.6.3.1.2 Matrix Spike/Matrix Spike Duplicate Summary (Form III VOA).
    - 2.6.3.1.3 Method Blank Summary (Form IV VOA): If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank, by instrument.

Exhibit B--Section 2  
Reporting Requirements and Order of Data Deliverables

- 2.6.3.1.4 GC/MS instrument performance check (Form V VOA): If more than a single form is necessary, forms shall be arranged in chronological order, by instrument.
- 2.6.3.1.5 Internal Standard Area and RT Summary (Form IX VOA): If more than a single form is necessary, forms shall be arranged in chronological order, by instrument.
- 2.6.3.2 Volatiles Sample Data. Sample data shall be arranged in packets with the Organic Analysis Data Sheets (Form I VOA, including Form I VOA-TIC), followed by the raw data for that volatile sample. These sample packets shall be placed in increasing EPA sample number order, considering both letters and numbers.
- 2.6.3.2.1 Target Compound Results, Organic Analysis Data Sheet (Form I VOA-1, VOA-2). Tabulated results (identification and quantitation) of the specified target compounds (Exhibit C, Volatiles) shall be included. The validation and release of these results are authorized by a specific, signed statement in the SDG Narrative (see Section 2.6.1). In the event that the laboratory manager cannot verify all data reported for each sample, the laboratory manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.
- 2.6.3.2.2 Tentatively Identified Compounds (Form I VOA-TIC). Form I VOA-TIC is the tabulated list of the highest probable match for up to 10 organic compounds that are not system monitoring compounds or internal standard compounds and are not listed in Exhibit C (Volatiles). It includes the Chemical Abstracts Service (CAS) registry number (if applicable), tentative identification, and estimated concentration. This form shall be included even if no compounds are found. If no compounds are found, indicate this on the form by entering "0" in the field for "Number Found."
- 2.6.3.2.3 Reconstructed Total Ion Chromatograms (for each sample or sample extract, including dilutions and reanalyses). Reconstructed ion chromatograms shall be normalized to the largest nonsolvent component and shall contain the following header information:
- EPA sample number,
  - Date and time of analysis,
  - GC/MS instrument identifier,

- Lab file identifier, and
  - Analyst ID.
- 2.6.3.2.3.1 Internal standards and system monitoring compounds shall be labeled with the names of compounds, either directly out from the peak or on a printout of retention times if retention times are printed over the peak.
- 2.6.3.2.3.2 If automated data system procedures are used for preliminary identification and/or quantitation of the target compounds, the complete data system report shall be included in all sample data packages, in addition to the reconstructed ion chromatogram. The complete data system report shall include all of the information listed below. For laboratories which do not use the automated data system procedures, a laboratory "raw data sheet" containing the following information shall be included in the sample data package, in addition to the chromatogram:
- EPA sample number,
  - Date and time of analysis,
  - Retention time or scan number of identified target compounds,
  - Ion used for quantitation with measured area,
  - Copy of area table from data system,
  - GC/MS instrument identifier,
  - Lab file identifier, and
  - Analyst ID.
- 2.6.3.2.3.3 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C (Volatiles), internal standards and system monitoring compounds.
- EICPs displaying each manual integration.
- 2.6.3.2.4 Other Required Information. For each sample, by each compound identified, the following items shall be included in the data package.
- Copies of raw spectra and copies of background-subtracted mass spectra of target compounds listed in Exhibit C (Volatiles) that are identified in the sample and corresponding background-subtracted target compound standard mass spectra. Spectra shall be labeled with EPA sample number, lab file identifier, and date and time of analysis, and GC/MS instrument identifier. Compound names shall be clearly marked on all spectra.
  - Copies of mass spectra of organic compounds not listed in Exhibit C with associated best-match spectra (minimum of one, maximum of three best matches). Spectra shall be labeled with EPA sample number, lab file identifier, date and time of analysis, and GC/MS instrument identifier. Compound names shall be clearly marked on all spectra.
- 2.6.3.3 Volatiles Standards Data

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- 2.6.3.3.1 Initial calibration data (Form VI VOA-1, VOA-2) shall be included in order by instrument, if more than one instrument is used.
- Volatile standard(s) reconstructed ion chromatograms and quantitation reports for the initial (five-point) calibration, labeled as in Section 2.6.3.2.3. Spectra are not required.
  - All initial calibration data that pertain to samples in the data package shall be included, regardless of when it was performed and for which Case. When more than one initial calibration is performed, the data shall be in chronological order, by instrument.
  - EICPs displaying each manual integration.
- 2.6.3.3.2 Initial calibration verification data (Form VII VOA-1, VOA-2) shall be included in order by instrument, if more than one instrument is used.
- Volatile standard(s) reconstructed ion chromatograms and quantitation reports for each initial calibration verification, labeled as in Section 2.6.3.2.3. Spectra are not required.
  - When more than one initial calibration verification is performed, forms shall be in chronological order, by instrument.
  - EICPs displaying each manual integration.
- 2.6.3.3.3 Continuing calibration data (Form VIII VOA-1, VOA-2) shall be included in order by instrument, if more than one instrument is used.
- Volatile standard(s) reconstructed ion chromatograms and quantitation reports for all continuing (12-hour) calibrations, labeled as in Section 2.6.3.2.3. Spectra are not required.
  - When more than one continuing calibration is performed, forms shall be in chronological order, by instrument.
  - EICPs displaying each manual integration.
- 2.6.3.3.4 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C (Volatiles), internal standards and system monitoring compounds.



2.6.3.4 Volatiles Raw QC Data

2.6.3.4.1 BFB data shall be arranged in chronological order by instrument for each 12-hour period, for each GC/MS system utilized.

- Bar graph spectrum, labeled as in Section 2.6.3.2.3.
- Mass listing, labeled as in Section 2.6.3.2.3.
- Reconstructed total ion chromatogram, labeled as in Section 2.6.3.2.3.

2.6.3.4.2 Blank data shall be arranged by type of blank (method, instrument and storage) and shall be in chronological order by instrument.

NOTE: This order is different from that used for samples.

- Tabulated results (Form I VOA-1, VOA-2).
- Tentatively identified compounds (Form I VOA-TIC) even if none are found.
- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.6.3.2.3.
- Target compound spectra with laboratory-generated standard, labeled as in Section 2.6.3.2.4. Data systems which are incapable of dual display shall provide spectra in the following order:
  - Raw target compound spectra.
  - Enhanced or background-subtracted spectra.
  - Laboratory-generated standard spectra.
- GC/MS library search spectra for tentatively identified compounds, labeled as in Section 2.6.3.2.4.
- Quantitation/calculation of tentatively identified compound concentrations.

2.6.3.4.3 Volatiles Matrix Spike Data

- Tabulated results (Form I VOA-1, VOA-2) of target compounds. Form I VOA-TIC is not required.
- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.6.3.2.3. Spectra are not required.

2.6.3.4.4 Volatiles Matrix Spike Duplicate Data

- Tabulated results (Form I VOA-1, VOA-2) of target compounds. Form I VOA-TIC is not required.
- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.6.3.2.3. Spectra are not required.

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2.6.4 Semivolatiles Data (modified OLM03.1)

2.6.4.1 Semivolatiles QC Summary

2.6.4.1.1 Surrogate Percent Recovery Summary (Form II SV).

2.6.4.1.2 Matrix Spike/Matrix Spike Duplicate Summary (Form III SV)

2.6.4.1.3 Method Blank Summary (Form IV SV): If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank, by instrument.

2.6.4.1.4 GC/MS Instrument Performance Check (Form V SV): If more than a single form is necessary, forms shall be arranged in chronological order, by instrument.

2.6.4.1.5 Internal Standard Area and RT Summary (Form IX SV): If more than a single form is necessary, forms shall be arranged in chronological order, by instrument.

2.6.4.2 Semivolatiles Sample Data. Sample data shall be arranged in packets with the Organic Analysis Data Sheet (Form I SV, including Form I SV-TIC), followed by the raw data for semivolatile samples. These sample packets shall be placed in increasing EPA sample number order, considering both letters and numbers.

2.6.4.2.1 Target Compound Results, Organic Analysis Data Sheet (Form I SV-1, SV-2). Tabulated results (identification and quantitation) of the specified target compounds (Exhibit C, Semivolatiles) shall be included. The validation and release of these results are authorized by a specific, signed statement in the SDG Narrative (see Section 2.6.1). In the event that the laboratory manager cannot verify all data reported for each sample, the laboratory manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

2.6.4.2.2 Semivolatile Tentatively Identified Compounds (Form I SV-TIC). Form I SV-TIC is the tabulated list of the highest probable match for up to 10 of the non-surrogate/non-internal standard organic compounds that are not listed in Exhibit C (Volatiles and Semivolatiles). It includes the CAS registry number (if applicable), tentative identification, and estimated concentration. This form shall be included even if no compounds are found. If no compounds are found, indicate this on the form by entering "0" in the field for "number found."

2.6.4.2.3 Reconstructed Total Ion Chromatograms (for each sample, including dilutions and reanalyses). Reconstructed ion chromatograms shall be normalized to the largest nonsolvent component and shall contain the following header information:

- EPA sample number,
- Date and time of analysis,
- GC/MS instrument identifier,
- Lab file identifier, and
- Analyst ID.

2.6.4.2.3.1 Internal standards and surrogate compounds shall be labeled with the names of compounds, either directly out from the peak or on a printout of retention times if retention times are printed over the peak.

2.6.4.2.3.2 If automated data system procedures are used for preliminary identification and/or quantitation of the target compounds, the complete data system report shall be included in all sample data packages, in addition to the reconstructed ion chromatogram. The complete data system report shall include all of the information listed below. For laboratories which

do not use the automated data system procedures, a laboratory "raw data sheet" containing the following information shall be included in the sample data package, in addition to the chromatogram.

- EPA sample number,
- Date and time of analysis,
- Retention time or scan number of identified target compounds,
- Ion used for quantitation with measured area,
- Copy of area table from data system,
- GC/MS instrument identifier, and
- Lab file identifier.

2.6.4.2.3.3 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C (Semivolatiles), internal standards and system monitoring compounds.

- EICPs displaying each manual integration.

2.6.4.2.4 Other Required Information. For each sample, by each compound identified, the following shall be included in the data package.

- Copies of raw spectra and copies of background-subtracted mass spectra of target compounds listed in Exhibit C (Semivolatiles) that are identified in the sample and corresponding background-subtracted target compound standard mass spectra. Spectra shall be labeled with EPA sample number, lab file identifier, and date and time of analysis, and GC/MS instrument identifier compound names shall be clearly marked on all spectra.
- Copies of mass spectra of non-surrogate/non-internal standard organic compounds not listed in Exhibit C (Volatiles and Semivolatiles) with associated best-match spectra (maximum of three best matches). This includes the mass spectra for tentatively identified alkanes. Spectra shall be labeled with EPA sample number, lab file identifier, and date and time of analysis, and GC/MS instrument identifier compound names shall be clearly marked on all spectra.

2.6.4.3 Semivolatiles Standards Data

2.6.4.3.1 Initial calibration data (Form VI SV-1, SV-2) shall be included in order by instrument, if more than one instrument used.

- Semivolatile standard(s) reconstructed ion chromatograms and quantitation reports for the initial (five-point) calibration, labeled as in Section 2.6.4.2.3. Spectra are not required.
- All initial calibration data that pertain to samples in the data package shall be included, regardless of when it was performed and for which Case. When more than one initial calibration is performed, the data shall be in chronological order, by instrument.
- EICPs displaying each manual integration.

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- 2.6.4.3.2 Initial calibration verification data (Form VII SV-1, SV-2) shall be included in order by instrument, if more than one instrument used.
- Semivolatile standard(s) reconstructed ion chromatograms and quantitation reports for each initial calibration verification, labeled as in Section 2.6.4.2.3. Spectra are not required.
  - When more than one initial calibration verification is performed, forms shall be in chronological order, by instrument.
  - EICPs displaying each manual integration.
- 2.6.4.3.3 Continuing calibration data (Form VIII SV-1, SV-2) shall be included in order by instrument, if more than one instrument used.
- Semivolatile standard(s) reconstructed ion chromatograms and quantitation reports for all continuing (12-hour) calibrations, labeled as in Section 2.6.4.2.3. Spectra are not required.
  - When more than one continuing calibration is performed, forms shall be in chronological order, by instrument.
  - EICPs displaying each manual integration.
- 2.6.4.3.4 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C (Semivolatiles), internal standards and system monitoring compounds.

2.6.4.4 Semivolatiles Raw QC Data

2.6.4.4.1 DFTPP data shall be arranged in chronological order by instrument for each 12-hour period, for each GC/MS system utilized.

- Bar graph spectrum, labeled as in Section 2.6.4.2.3.
- Mass listing, labeled as in Section 2.6.4.2.3.
- Reconstructed total ion chromatogram, labeled as in Section 2.6.4.2.3.

2.6.4.4.2 Blank data shall be included in chronological order by extraction date.

NOTE: This order is different from that used for samples.

- Tabulated results (Form I SV-1, SV-2).
- Tentatively identified compounds (Form I SV-TIC) even if none are found.
- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.6.4.2.3.
- Target compound spectra with laboratory-generated standard, labeled as in Section 2.6.4.2.4. Data systems which are incapable of dual display shall provide spectra in the following order:
  - Raw target compound spectra.
  - Enhanced or background-subtracted spectra.
  - Laboratory-generated standard spectra.
- GC/MS library search spectra for tentatively identified compounds, labeled as in Section 2.6.4.2.4.
- Quantitation/calculation of tentatively identified compound concentrations.

2.6.4.4.3 Semivolatiles Matrix Spike Data

- Tabulated results (Form I SV-1, SV-2) of target compounds. Form I SV-TIC is not required.
- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.6.4.2.3. Spectra are not required.

2.6.4.4.4 Semivolatiles Matrix Spike Duplicate Data

- Tabulated results (Form I SV-1, SV-2) of target compounds. Form I SV-TIC is not required.
- Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.6.4.2.3. Spectra are not required.

2.6.4.4.5 Semivolatile GPC Data. The UV traces for the GPC calibration solution and the reconstructed ion chromatogram and data system reports for the GPC blank shall be arranged in chronological order by GPC for the GPC calibration.

- UV traces labeled with the GPC column identifier, date of calibration, and compound names. Compound names shall be placed directly out from the peak, or on the printout of retention times when the retention times are printed directly over the peak.

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- Reconstructed ion chromatogram and data system report(s) labeled as specified in Section 2.6.4.2.3 for GPC blank analysis.
- Reconstructed ion chromatogram and data system report(s) for all standards used to quantify compounds in the GPC blank labeled as specified in Section 2.6.4.2.3.

2.6.5 Pesticide/Aroclor Data (modified OLM03.1)

2.6.5.1 Pesticide/Aroclor QC Summary

2.6.5.1.1 Surrogate Percent Recovery Summary (Form II PEST).

2.6.5.1.2 Matrix Spike/Matrix Spike Duplicate Summary (Form III PEST).

2.6.5.1.3 Method Blank Summary (Form IV PEST): If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank.

2.6.5.2 Pesticide/Aroclor Sample Data. Sample data shall be arranged in packets with the Organic Analysis Data Sheet (Form I PEST), followed by the raw data for pesticide samples. These sample packets should then be placed in increasing EPA sample number order, considering both letters and numbers.

2.6.5.2.1 Target Compound Results, Organic Analysis Data Sheet (Form I PEST). Tabulated results (identification and quantitation) of the specified target compounds (Exhibit C, Pesticides/Aroclors) shall be included. The validation and release of these results is authorized by a specific, signed statement in the SDG Narrative (see Section 2.6.1). In the event that the laboratory manager cannot verify all data reported for each sample, the laboratory manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.

2.6.5.2.2 Copies of Pesticide Chromatograms. Positively identified compounds shall be labeled with the names of compounds, either directly out from the peak on the chromatogram, or on a printout of retention times on the data system printout if retention times are printed over the peak on the chromatogram. All chromatograms shall meet the acceptance criteria in Exhibit D PEST, and shall be labeled with the following information:

- EPA sample number,
- Volume injected ( $\mu$ L),
- Date and time of injection,
- GC column identifier (by stationary phase and internal diameter),
- GC instrument identifier, and
- Scaling factor.

2.6.5.2.3 Copies of pesticide chromatograms from second GC column shall be included and labeled as in Section 2.6.5.2.2.

2.6.5.2.4 Data System Printout. A printout of retention time and corresponding peak height or peak area shall accompany each chromatogram. The printout shall be labeled with the EPA sample number. In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/EC operator must identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration time range.

2.6.5.2.5 All manual work sheets shall be included in the sample data package.

- 2.6.5.2.6 Other Required Information. If pesticides/Aroclors are confirmed by GC/MS, the Contractor shall submit copies of reconstructed ion chromatograms, raw spectra and background-subtracted mass spectra of target compounds listed in Exhibit C (Pesticides/Aroclors) that are identified in the sample and corresponding background-subtracted TCL standard mass spectra. Compound names shall be clearly marked on all spectra. For multicomponent pesticides/Aroclors confirmed by GC/MS, the Contractor shall submit mass spectra of three major peaks of multicomponent compounds from samples and standards.
- 2.6.5.3 Pesticide/Aroclor Standards Data
- 2.6.5.3.1 Initial Calibration of Single Component Analytes (Form VI PEST-1, PEST-2): for all GC columns, all instruments, in chronological order by GC column and instrument.
- 2.6.5.3.2 Initial Calibration of Multicomponent Analytes (Form VI PEST-3, PEST-4): for all GC columns, all instruments, in chronological order by GC column and instrument.
- 2.6.5.3.3 Analyte Resolution Summary (Form VI PEST-5): for all GC columns and instruments, in chronological order by GC column and instrument.
- 2.6.5.3.4 Performance Evaluation Mixture (Form VI PEST-6): for all GC columns and instruments, in chronological order by GC column and instrument.
- 2.6.5.3.5 Individual Standard Mixture A (Form VI PEST-7): for all GC columns and instruments, in chronological order by GC column and instrument.
- 2.6.5.3.6 Individual Standard Mixture B (Form VI PEST-8): for all GC columns and instruments, in chronological order by GC column and instrument.
- 2.6.5.3.7 Calibration Verification Summary (Form VIII PEST-1): for all performance evaluation mixtures and instrument blanks, on all GC columns and instruments, in chronological order by GC column and instrument.
- 2.6.5.3.8 Calibration Verification Summary (Form VIII PEST-2): for all mid-point concentrations of Individual Standard Mixtures A and B and instrument blanks used for calibration verification, on all GC columns and instruments, in chronological order by GC column and instrument.
- 2.6.5.3.9 Analytical Sequence (Form IX PEST): for all GC columns and instruments, in chronological order by GC column and instrument.
- 2.6.5.3.10 Florisil Cartridge Check (Form X PEST-1): for all lots of cartridges used to process samples in the SDG.
- 2.6.5.3.11 Pesticide GPC Calibration (Form X PEST-2): for all GPC columns, in chronological order by calibration date.
- 2.6.5.3.12 Pesticide Identification Summary for Single Component Analytes (Form XI PEST-1): for all samples with positively identified single component analytes, in order by increasing EPA sample number.
- 2.6.5.3.13 Pesticide Identification Summary for Multicomponent Analytes (Form XI PEST-2): for all samples with positively identified multicomponent analytes, in order by increasing EPA sample number.
- 2.6.5.3.14 Chromatograms and data system printouts shall be included for all standards including the following:
- Resolution check mixture.

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- Performance evaluation mixtures, all.
- Individual Standard Mixture A, at three concentrations, each initial calibration.
- Individual Standard Mixture B, at three concentrations, each initial calibration.
- All multicomponent analytes (toxaphene and Aroclors), each initial calibration.
- All mid-point concentrations of Individual Standard Mixtures A and B used for continuing calibration.
- All multicomponent analyte standards analyzed for confirmation.

2.6.5.3.15 A printout of retention time and corresponding peak height or peak area shall accompany each chromatogram. The printout shall be labeled with the EPA sample number. In addition, all chromatograms shall meet the acceptance criteria in Exhibit D PEST, and shall be labeled with the following:

- EPA sample number for the standard, e.g., INDA1, INDA2, etc. (See Section 4 for details.)
- Label all standard peaks for all individual compounds either directly out from the peak on the chromatogram or on the printout of retention times on the data system printout if retention times are printed over the peak on the chromatogram.
- Total nanograms injected for each standard. When total nanograms injected appear on the printout, it is not necessary to include them on the chromatogram.
- Date and time of injection.
- GC column identifier (by stationary phase and internal diameter).
- GC instrument identifier.
- Scaling factor.

NOTE: In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/EC operator must identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration time range.

2.6.5.4 Pesticide/Aroclor Raw QC Data

2.6.5.4.1 Blank data shall be arranged by type of blank (method, instrument, sulfur cleanup, sulfuric acid cleanup) and shall be in chronological order by instrument.

NOTE: This order is different from that used for samples.

- Tabulated results (Form I PEST).
- Chromatogram(s) and data system printout(s) (GC) for each GC column and instrument used for analysis, labeled as in Sections 2.6.5.2.2 and 2.6.5.2.4.

2.6.5.4.2 Matrix Spike Data

- Tabulated results (Form I PEST) of target compounds.
- Chromatogram(s) and data system printout(s) (GC), labeled as in Sections 2.6.5.2.2 through 2.6.5.2.4.

2.6.5.4.3 Matrix Spike Duplicate Data



- Tabulated results (Form I PEST) of target compounds.
  - Chromatogram(s) and data system printout(s) (GC), labeled as in Sections 2.6.5.2.2 through 2.6.5.2.4.
- 2.6.5.5 Raw GPC Data
- 2.6.5.5.1 GPC Calibration. The UV traces for the GPC calibration solution, chromatograms, and the data system reports for the GPC blank shall be arranged in chronological order for the GPC calibration.
- UV traces labeled with the GPC column identifier, date of calibration, and compound names. Compound names shall be placed directly out from the peak, or on the printout of retention times when the retention times are printed directly over the peak.
  - Chromatogram and data system report(s) labeled as specified in Sections 2.6.5.2.2 and 2.6.5.2.4 for GPC blank analysis.
  - Chromatogram and data system report(s) for all standards used to quantify compounds in the GPC blank labeled as specified in Section 2.6.5.3.16 (i.e., Individual Standard Mixture A, Individual Standard Mixture B, and the Aroclor/toxaphene standards).
- 2.6.5.5.2 GPC Calibration Check. The chromatogram and the data system report(s) shall be arranged in chronological order for the GPC calibration check.
- Chromatograms and data system printouts labeled as specified in Sections 2.6.5.2.2 and 2.6.5.2.4 for the GPC calibration check solution analyses.
  - Chromatogram and data system report(s) for standards used to quantify compounds in the GPC calibration check solution or used to assess the Aroclor pattern labeled as specified in Section 2.6.5.3.15 (i.e., Individual Standard Mixtures A and B and Aroclor Standard Mixture 1016/1260 form the initial calibration sequence).
- 2.6.5.6 Raw Florisil Data. The chromatogram and data system report(s) shall be arranged in chronological order by Florisil cartridge performance check analyses.
- Chromatograms and data system reports labeled as specified in Sections 2.6.5.2.2 and 2.6.5.2.4 for the florisil cartridge performance check analyses.
  - Chromatograms and data system reports for standard analyses used to quantify compounds in the Florisil cartridge performance check analysis, labeled as specified in Section 2.6.5.3.15 (i.e., Individual Standard Mixture A and Individual Standard Mixture B and the 2,4,5 Trichlorophenol solution).
- 2.6.6 EPA New England Modified 524.2 Volatiles Data
- 2.6.6.1 Modified 524.2 Volatiles QC Summary
- 2.6.6.1.1 System Monitoring Compound Summary (Form II 524.2).
- 2.6.6.1.2 Matrix Spike/Matrix Spike Duplicate Summary (Form III 524.2).
- 2.6.6.1.3 Method Blank Summary (Form IV 524.2): If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank, by instrument.
- 2.6.6.1.4 GC/MS instrument performance check (Form V 524.2): If more than a single form is necessary, forms shall be arranged in chronological order, by instrument.

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- 2.6.6.1.5 Internal Standard Area and RT Summary (Form IX 524.2): If more than a single form is necessary, forms shall be arranged in chronological order, by instrument.
- 2.6.6.2 Modified 524.2 Volatiles Sample Data. Sample data shall be arranged in packets with the Organic Analysis Data Sheet (Form I 524.2, including Form I 524.2-TIC), followed by the raw data for volatile samples. These sample packets shall be placed in increasing EPA sample number order, considering both letters and numbers.
- 2.6.6.2.1 Target Compound Results, Organic Analysis Data Sheet (Form I 524.2-1, 524.2-2). Tabulated results (identification and quantitation) of the specified target compounds (Exhibit C, 524.2 Volatiles) shall be included. The validation and release of these results are authorized by a specific, signed statement in the SDG Narrative (see Section 2.6.1). In the event that the laboratory manager cannot verify all data reported for each sample, the laboratory manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.
- 2.6.6.2.2 Tentatively Identified Compounds (Form I 524.2-TIC). Form I 524.2-TIC is the tabulated list of the highest probable match for up to 10 organic compounds that are not system monitoring compounds or internal standard compounds and are not listed in Exhibit C. It includes the Chemical Abstracts Service (CAS) registry number (if applicable), tentative identification, and estimated concentration. This form shall be included even if no compounds are found. If no compounds are found, indicate this on the form by entering "0" in the field for "Number Found."
- 2.6.6.2.3 Reconstructed Total Ion Chromatograms (for each sample or sample extract, including dilutions and reanalyses). Reconstructed ion chromatograms shall be normalized to the largest nonsolvent component and shall contain the following header information:
- EPA sample number,
  - Date and time of analysis,
  - GC/MS instrument identifier,
  - Lab file identifier, and
  - Analyst ID.
- 2.6.6.2.3.1 Internal standards and system monitoring compounds shall be labeled with the names of compounds, either directly out from the peak or on a printout of retention times if retention times are printed over the peak.
- 2.6.6.2.3.2 If automated data system procedures are used for preliminary identification and/or quantitation of the target compounds, the complete data system report shall be included in all sample data packages, in addition to the reconstructed ion chromatogram. The complete data system report shall include all of the information listed below. For laboratories which do not use the automated data system procedures, a laboratory "raw data sheet" containing the following information shall be included in the sample data package, in addition to the chromatogram:
- EPA sample number,
  - Date and time of analysis,
  - Retention time or scan number of identified target compounds,
  - Ion used for quantitation with measured area,

- Copy of area table from data system,
- GC/MS instrument identifier,
- Lab file identifier, and
- Analyst ID.

2.6.6.2.3.3 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C (524.2 Volatiles), internal standards and system monitoring compounds.

- EICPs displaying each manual integration.

2.6.6.2.4 Other Required Information. For each sample, by each compound identified, the following items shall be included in the data package.

- Copies of raw spectra and copies of background-subtracted mass spectra of target compounds listed in Exhibit C (524.2 Volatiles) that are identified in the sample and corresponding background-subtracted target compound standard mass spectra. Spectra shall be labeled with EPA sample number, lab file identifier, and date and time of analysis, and GC/MS instrument identifier. Compound names shall be clearly marked on all spectra.
- Copies of mass spectra of organic compounds not listed in Exhibit C with associated best-match spectra (minimum of one, maximum of three best matches). Spectra shall be labeled with EPA sample number, lab file identifier, and date and time of analysis, and GC/MS instrument identifier. Compound names shall be clearly marked on all spectra.

2.6.6.3 Modified 524.2 Volatiles Standards Data

2.6.6.3.1 Initial calibration data (Form VI 524.2-1, 524.2-2) shall be included in order by instrument, if more than one instrument is used.

- Volatile standard(s) reconstructed ion chromatograms and quantitation reports for the initial (five-point) calibration, labeled as in Section 2.6.6.2.3. Spectra are not required.
- All initial calibration data that pertain to samples in the data package shall be included, regardless of when it was performed and for which Case. When more than one initial calibration is performed, the data shall be in chronological order, by instrument.
- EICPs displaying each manual integration.

2.6.6.3.2 Initial calibration verification data (Form VII 524.2-1, 524.2-2) shall be included in order by instrument, if more than one instrument is used.

- Volatile standard(s) reconstructed ion chromatograms and quantitation reports for each initial calibration verification, labeled as in Section 2.6.6.2.3. Spectra are not required.
- When more than one initial calibration verification is performed, forms shall be in chronological order, by instrument.

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- EICPs displaying each manual integration.
- 2.6.6.3.3 Continuing calibration data (Form VIII 524.2-1, 524.2-2) shall be included in order by instrument, if more than one instrument is used.
- Volatile standard(s) reconstructed ion chromatograms and quantitation reports for all continuing (12-hour) calibrations, labeled as in Section 2.6.6.2.3. Spectra are not required.
  - When more than one continuing calibration is performed, forms shall be in chronological order, by instrument.
  - EICPs displaying each manual integration.
- 2.6.6.3.4 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS operator shall identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration scan range. In addition, a hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all compounds listed in Exhibit C (524.2 Volatiles), internal standards and system monitoring compounds.
- 2.6.6.4 Modified 524.2 Volatiles Raw QC Data
- 2.6.6.4.1 BFB data shall be arranged in chronological order by instrument for each 12-hour period, for each GC/MS system utilized.
- Bar graph spectrum, labeled as in Section 2.6.6.2.3.
  - Mass listing, labeled as in Section 2.6.6.2.3.
  - Reconstructed total ion chromatogram, labeled as in Section 2.6.6.2.3.
- 2.6.6.4.2 Blank data shall be arranged by type of blank (method, storage, instrument) and shall be in chronological order by instrument.
- NOTE: This order is different from that used for samples.
- Tabulated results (Form I 524.2-1, 524.2-2).
  - Tentatively identified compounds (Form I 524.2-TIC) even if none are found.
  - Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.6.6.2.3.
  - Target compound spectra with laboratory-generated standard, labeled as in Section 2.6.6.2.4. Data systems which are incapable of dual display shall provide spectra in the following order:
    - Raw target compound spectra.
    - Enhanced or background-subtracted spectra.
    - Laboratory-generated standard spectra.
  - GC/MS library search spectra for tentatively identified compounds, labeled as in Section 2.6.6.2.4.
  - Quantitation/calculation of tentatively identified compound concentrations.

- 2.6.6.4.3      Modified 524.2 Volatiles Matrix Spike Data
- Tabulated results (Form I 524.2-1, 524.2-2) of target compounds. Form I 524.2-TIC is not required.
  - Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.6.6.2.3. Spectra are not required.
- 2.6.6.4.4      Modified 524.2 Volatiles Matrix Spike Duplicate Data
- Tabulated results (Form I 524.2-1, 524.2-2) of target compounds. Form I 524.2-TIC is not required.
  - Reconstructed ion chromatogram(s) and quantitation report(s), labeled as in Section 2.6.6.2.3. Spectra are not required.
- 2.6.7      Aroclor Data (PCBs only, modified 8080)
- 2.6.7.1      Aroclor QC Summary
- 2.6.7.1.1      Surrogate Percent Recovery Summary (Form II PCB).
- 2.6.7.1.2      Matrix Spike/Matrix Spike Duplicate Summary (Form III PCB).
- 2.6.7.1.3      Method Blank Summary (Form IV PCB): If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank.
- 2.6.7.2      Aroclor Sample Data. Sample data shall be arranged in packets with the Organic Analysis Data Sheet (Form I PCB), followed by the raw data for the Aroclor samples. These sample packets should then be placed in increasing EPA sample number order, considering both letters and numbers.
- 2.6.7.2.1      Target Compound Results, Organic Analysis Data Sheet (Form I PCB). Tabulated results (identification and quantitation) of the specified target Aroclors (Exhibit C, Aroclors) shall be included. The validation and release of these results is authorized by a specific, signed statement in the SDG Narrative (see Section 2.6.1). In the event that the laboratory manager cannot verify all data reported for each sample, the laboratory manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.
- 2.6.7.2.2      Copies of Aroclor Chromatograms. Positively identified Aroclors shall be labeled with the name of the Aroclor, either on the chromatogram directly out from each peak in the set of 3 to 5 peaks chosen for calibration, or on a printout of retention times on the data system printout if retention times are printed over the peaks on the chromatogram. All chromatograms shall meet the acceptance criteria in Exhibit D PCB, and shall be labeled with the following information:
- EPA sample number,
  - Volume injected ( $\mu$ L),
  - Date and time of injection,
  - GC column identifier (by stationary phase and internal diameter),

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- GC instrument identifier, and
  - Scaling factor.
- 2.6.7.2.3 Copies of Aroclor chromatograms for the second GC column shall be included and labeled as in Section 2.6.7.2.2.
- 2.6.7.2.4 Data System Printout. A printout of retention time and corresponding peak height or peak area shall accompany each chromatogram. The printout shall be labeled with the EPA sample number. In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/EC operator must identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration time range.
- 2.6.7.2.5 All manual work sheets shall be included in the sample data package.
- 2.6.7.2.6 Other Required Information. If Aroclors are confirmed by GC/MS, the Contractor shall submit copies of the reconstructed ion chromatograms, raw spectra and background-subtracted mass spectra of three major peaks of the multicomponent compounds listed in Exhibit C (Aroclors) that are identified in the sample and background-subtracted TCL standard mass spectra. Compound names shall be clearly marked on all spectra.
- 2.6.7.3 Aroclor Standards Data
- 2.6.7.3.1 Initial Calibration of Aroclors (Form VI PCB-1, PCB-2): for all GC columns, all instruments, in chronological order by GC column and instrument.
- 2.6.7.3.2 Aroclor Resolution Summary (Form VI PCB-3): for all GC columns and instruments, in chronological order by GC column and instrument.
- 2.6.7.3.3 Continuing Calibration Summary (Form VIII PCB): for all mid-level Aroclor standards (1254 and/or additional Aroclors determined present for that SDG) used for continuing calibration on all GC columns and instruments, in chronological order by GC column and instrument.
- 2.6.7.3.4 Analytical Sequence (Form IX PCB): for all GC columns and instruments, in chronological order by GC column and instrument.
- 2.6.7.3.5 Aroclor Identification Summary for Multicomponent Analytes (Form XI PCB): for all samples with positively identified multicomponent analytes, in order by increasing EPA sample number.
- 2.6.7.3.6 Chromatograms and data system printouts shall be included for all standards including the following:
- All multicomponent Aroclors for the initial calibration.
  - All mid-point concentration Aroclors used for continuing calibration.
  - All multicomponent Aroclor standards analyzed for confirmation.
- 2.6.7.3.7 A printout of retention time and corresponding peak height or peak area shall accompany each chromatogram. The printout shall be labeled with the EPA sample number. In addition, all chromatograms shall meet the acceptance criteria in Exhibit D PCB, and shall be labeled with the following:
- EPA sample number for each standard, e.g., AR1260L, AR1260M, etc. (See Section 3 for details.)

- Label all standard peaks associated with each Aroclor either directly out from the peak on the chromatogram or on the printout of retention times on the data system printout if retention times are printed over the peak on the chromatogram.
- Total nanograms injected for each standard. When total nanograms injected appear on the printout, it is not necessary to include them on the chromatogram.
- Date and time of injection.
- GC column identifier (by stationary phase and internal diameter).
- GC instrument identifier.
- Scaling factor.

NOTE: In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/EC operator must identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration time range.

2.6.7.4 Aroclor Raw QC Data

- 2.6.7.4.1 Blank data shall be arranged by type of blank (method, instrument, sulfur cleanup) and shall be in chronological order by instrument.

NOTE: This order is different from that used for samples.

- Tabulated results (Form I PCB).
- Chromatogram(s) and data system printout(s) (GC) for each GC column and instrument used for analysis, labeled as in Sections 2.6.7.2.2 and 2.6.7.2.4.

2.6.7.4.2 Matrix Spike Data

- Tabulated results (Form I PCB) of target Aroclors.
- Chromatogram(s) and data system printout(s) (GC), labeled as in Sections 2.6.7.2.2 through 2.6.7.2.4.

2.6.7.4.3 Matrix Spike Duplicate Data

- Tabulated results (Form I PCB) of target Aroclors.
- Chromatogram(s) and data system printout(s) (GC), labeled as in Sections 2.6.7.2.2 through 2.6.7.2.4.

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- 2.6.8 Water Soluble Organics - GC/NPD Data (modified 8015A)
- 2.6.8.1 Water Soluble Organics - GC/NPD QC Summary
- 2.6.8.1.1 Surrogate Percent Recovery Summary (Form II WSO/NPD).
- 2.6.8.1.2 Matrix Spike/Matrix Spike Duplicate Summary (Form III WSO/NPD).
- 2.6.8.1.3 Method Blank Summary (Form IV WSO/NPD): If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank.
- 2.6.8.2 Water Soluble Organics - GC/NPD Sample Data. Sample data shall be arranged in packets with the Organic Analysis Data Sheets (Form I WSO/NPD), followed by the raw data for the samples. These sample packets should then be placed in increasing EPA sample number order, considering both letters and numbers.
- 2.6.8.2.1 Target Compound Results, Organic Analysis Data Sheet (Form I WSO/NPD). Tabulated results (identification and quantitation) of the specified target compounds (Exhibit C, Water Soluble Organics - GC/NPD) shall be included. The validation and release of these results is authorized by a specific, signed statement in the SDG Narrative (see Section 2.6.1). In the event that the laboratory manager cannot verify all data reported for each sample, the laboratory manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.
- 2.6.8.2.2 Copies of Water Soluble Organics - GC/NPD Chromatograms. Positively identified compounds shall be labeled with the names of compounds, either directly out from the peak on the chromatogram, or on a printout of retention times on the data system printout if retention times are printed over the peak on the chromatogram. All chromatograms shall meet the acceptance criteria in Exhibit D, WSO/NPD, and shall be labeled with the following information:
- EPA sample number,
  - Volume injected ( $\mu$ L),
  - Date and time of injection,
  - GC column identifier (by stationary phase and internal diameter),
  - GC instrument identifier, and
  - Scaling factor.
- 2.6.8.2.3 Copies of GC/NPD chromatograms from the second GC column shall be included and labeled as in Section 2.6.9.2.2.
- 2.6.8.2.4 Data System Printout. A printout of retention time and corresponding peak height or peak area shall accompany each chromatogram. The printout shall be labeled with the EPA sample number. In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/NPD operator must identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration time range.
- 2.6.8.2.5 All manual work sheets shall be included in the sample data package.
- 2.6.8.2.6 Other Required Information. If the water soluble compounds are confirmed by GC/MS, the Contractor shall submit copies of reconstructed ion chromatograms, raw spectra and background-subtracted mass spectra of target compounds listed in Exhibit C (Water Soluble Organics - GC/NPD) that are identified in the sample and corresponding background-subtracted TCL standard



mass spectra. Compound names shall be clearly marked on all spectra.

2.6.8.3 Water Soluble Organics - GC/NPD Standards Data

- 2.6.8.3.1 Initial Calibration of the Water Soluble Organics - GC/NPD (Form VI WSO/NPD-1): for all GC columns, all instruments, in chronological order by GC column and instrument.
- 2.6.8.3.2 Water Soluble Organics - GC/NPD Resolution Summary (Form VI WSO/NPD-2): for all GC columns and instruments, in chronological order by GC column and instrument.
- 2.6.8.3.3 Initial Calibration Verification Summary (Form VII GC/NPD): for all second source mid-point standards used for initial calibration verification, on all GC columns and instruments, in chronological order by GC column and instrument.
- 2.6.8.3.4 Continuing Calibration Summary (Form VIII WSO/NPD): for all mid-point standards used for calibration verification, on all GC columns and instruments, in chronological order by GC column and instrument.
- 2.6.8.3.5 Analytical Sequence (Form IX WSO/NPD): for all GC columns and instruments, in chronological order by GC column and instrument.
- 2.6.8.3.6 Water Soluble Organics - GC/NPD Identification Summary (Form XI WSO/NPD): for all samples with positively identified analytes, in order by increasing EPA sample number.
- 2.6.8.3.7 Chromatograms and data system printouts shall be included for all standards including the following:
- All individual calibration mixtures, at three concentration levels, for each initial calibration.
  - All second source mid-level standards used for initial calibration verification.
  - All mid-level standards used for calibration verification.
- 2.6.8.3.8 A printout of retention time and corresponding peak height or peak area shall accompany each chromatogram. The printout shall be labeled with the EPA sample number. In addition, all chromatograms shall meet the acceptance criteria in Exhibit D, WSO/NPD, and shall be labeled with the following:
- EPA sample number for each standard (See Section 3 for details.).
  - Label all standard peaks associated with each analyte either directly out from the peak on the chromatogram or on the printout of retention times on the data system printout if retention times are printed over the peak on the chromatogram.
  - Date and time of injection.
  - GC column identifier (by stationary phase and internal diameter).
  - GC instrument identifier.
  - Scaling factor.
- NOTE: In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/NPD operator must identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration time range.

2.6.8.4 Water Soluble Organics - GC/NPD Raw QC Data

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- 2.6.8.4.1 Blank data shall be arranged by type of blank (method, instrument) and shall be in chronological order by instrument.
- NOTE: This order is different from that used for samples.
- Tabulated results (Form I WSO/NPD).
  - Chromatogram(s) and data system printout(s) (GC) for each GC column and instrument used for analysis, labeled as in Sections 2.6.9.2.2 and 2.6.9.2.4.
- 2.6.8.4.2 Matrix Spike Data
- Tabulated results (Form I WSO/NPD) of target analytes.
  - Chromatogram(s) and data system printout(s) (GC), labeled as in Sections 2.6.9.2.2 through 2.6.9.2.4.
- 2.6.8.4.3 Matrix Spike Duplicate Data
- Tabulated results (Form I WSO/NPD) of target analytes.
  - Chromatogram(s) and data system printout(s) (GC), labeled as in Sections 2.6.9.2.2 through 2.6.9.2.4.
- 2.6.9 Water Soluble Organics - GC/FID Data (modified 8015A)
- 2.6.9.1 Water Soluble Organics - GC/FID QC Summary
- 2.6.9.1.1 Surrogate Percent Recovery Summary (Form II WSO/FID).
- 2.6.9.1.2 Matrix Spike/Matrix Spike Duplicate Summary (Form III WSO/FID).
- 2.6.9.1.3 Method Blank Summary (Form IV WSO/FID): If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank.
- 2.6.9.2 Water Soluble Organics - GC/FID Sample Data. Sample data shall be arranged in packets with the Organic Analysis Data Sheets (Form I WSO/FID), followed by the raw data for the samples. These sample packets should then be placed in increasing EPA sample number order, considering both letters and numbers.
- 2.6.9.2.1 Target Compound Results, Organic Analysis Data Sheet (Form I WSO/FID). Tabulated results (identification and quantitation) of the specified target compounds (Exhibit C, Water Soluble Organics - GC/FID) shall be included. The validation and release of these results is authorized by a specific, signed statement in the SDG Narrative (see Section 2.6.1). In the event that the laboratory manager cannot verify all data reported for each sample, the laboratory manager shall provide a detailed description of the problems associated with the sample in the SDG Narrative.
- 2.6.9.2.2 Copies of Water Soluble Organics - GC/FID Chromatograms. Positively identified compounds shall be labeled with the names of compounds, either directly out from the peak on the chromatogram, or on a printout of retention times on the data system printout if retention times are printed over the peak on the chromatogram. All chromatograms shall meet the acceptance criteria in Exhibit D, WSO/FID, and shall be labeled with the following information:
- EPA sample number,
  - Volume injected ( $\mu$ L),
  - Date and time of injection,
  - GC column identifier (by stationary phase and internal diameter),
  - GC instrument identifier, and

- Scaling factor.
- 2.6.9.2.3 Copies of GC/FID chromatograms from the second GC column shall be included and labeled as in Section 2.6.10.2.2.
- 2.6.9.2.4 Data System Printout. A printout of retention time and corresponding peak height or peak area shall accompany each chromatogram. The printout shall be labeled with the EPA sample number. In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/FID operator must identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration time range.
- 2.6.9.2.5 All manual work sheets shall be included in the sample data package.
- 2.6.9.2.6 Other Required Information. If the water soluble compounds are confirmed by GC/MS, the Contractor shall submit copies of reconstructed ion chromatograms, raw spectra and background-subtracted mass spectra of target compounds listed in Exhibit C (Water Soluble Organics - GC/FID) that are identified in the sample and corresponding background-subtracted TCL standard mass spectra. Compound names shall be clearly marked on all spectra.
- 2.6.9.3 Water Soluble Organics - GC/FID Standards Data
- 2.6.9.3.1 Initial Calibration of the Water Soluble Organics - GC/FID (Form VI WSO/FID-1): for all GC columns, all instruments, in chronological order by GC column and instrument.
- 2.6.9.3.2 Water Soluble Organics GC/FID Resolution Summary (Form VI WSO/FID-2): for all GC columns and instruments, in chronological order by GC column and instrument.
- 2.6.9.3.3 Initial Calibration Verification Summary (Form VII GC/NPD): for all second source mid-point standards used for initial calibration verification, on all GC columns and instruments, in chronological order by GC column and instrument.
- 2.6.9.3.4 Continuing Calibration Summary (Form VIII WSO/FID): for all mid-point standards used for calibration verification, on all GC columns and instruments, in chronological order by GC column and instrument.
- 2.6.9.3.5 Analytical Sequence (Form IX WSO/FID): for all GC columns and instruments, in chronological order by GC column and instrument.
- 2.6.9.3.6 Water Soluble Organics - GC/FID Identification Summary (Form XI WSO/FID): for all samples with positively identified analytes, in order by increasing EPA sample number.
- 2.6.9.3.7 Chromatograms and data system printouts shall be included for all standards including the following:
- All individual calibration mixtures, at three concentration levels, for each initial calibration.
  - All second source mid-level standards used for initial calibration verification.
  - All mid-level standards used for calibration verification.
- 2.6.9.3.8 A printout of retention time and corresponding peak height or peak area shall accompany each chromatogram. The printout shall be labeled with the EPA sample number. In addition, all chromatograms shall meet the acceptance criteria in Exhibit D, WSO/FID, and shall be labeled with the following:

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- EPA sample number for each standard (See Section 3 for details.).
- Label all standard peaks associated with each analyte either directly out from the peak on the chromatogram or on the printout of retention times on the data system printout if retention times are printed over the peak on the chromatogram.
- Date and time of injection.
- GC column identifier (by stationary phase internal diameter).
- GC instrument identifier.
- Scaling factor.

NOTE: In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/FID operator must identify such edits or manual procedures by initialing and dating the changes made to the report, and shall include the integration time range.

2.6.9.4 Water Soluble Organics - GC/FID Raw QC Data

2.6.9.4.1 Blank data shall be arranged by type of blank (method, instrument) and shall be in chronological order by instrument.

NOTE: This order is different from that used for samples.

- Tabulated results (Form I WSO/FID).
- Chromatogram(s) and data system printout(s) (GC) for each GC column and instrument used for analysis, labeled as in Sections 2.6.10.2.2 and 2.6.10.2.4.

2.6.9.4.2 Matrix Spike Data

- Tabulated results (Form I WSO/FID) of target analytes.
- Chromatogram(s) and data system printout(s) (GC), labeled as in Sections 2.6.10.2.2 through 2.6.10.2.4.

2.6.9.4.3 Matrix Spike Duplicate Data

- Tabulated results (Form I WSO/FID) of target analytes.
- Chromatogram(s) and data system printout(s) (GC), labeled as in Sections 2.6.10.2.2 through 2.6.10.2.4.

2.7 Complete SDG File. As specified in Section 1, the Contractor shall deliver one Complete SDG File (CSF), including the original sample data package, to the Regional Sample Control Center (RSCC).

2.7.1 The CSF will contain all original documents specified in Sections 3 and 4 and in Form DC-2 (see Section 4). No photocopies of original documents will be placed in the CSF unless the original data was initially written in a bound notebook, maintained by the Contractor, or the originals were previously submitted to the Agency with another Case/SDG in accordance with the requirements described in Exhibit F. The contents of the CSF shall be numbered according to the specifications described in Section 3.25.

2.7.2 The CSF will consist of the following original documents in addition to the documents in the sample data package.

NOTE: All SDG-related documentation may be used or admitted as evidence in subsequent legal proceedings. Any other SDG-specific documents generated after the CSF is sent to the EPA, as well as copies that are altered in any fashion, are also deliverables to the EPA.

- 2.7.2.1 The original sample data package.
- 2.7.2.2 A completed and signed document inventory sheet (Form DC-2).
- 2.7.2.3 All original shipping documents including, but not limited to, the following documents:
  - EPA Chain-of-Custody Record,
  - Airbills,
  - EPA Traffic Reports, and
  - Sample tags (if present) sealed in plastic bags.
- 2.7.2.4 All original receiving documents including, but not limited to, the following documents:
  - Form DC-1,
  - Other receiving forms or copies of receiving logbooks, and
  - SDG cover sheet.
- 2.7.2.5 All original laboratory records, not already submitted in the sample data package, of sample transfer, preparation and analysis including, but not limited to, the following documents:
  - Original preparation and analysis forms or copies of preparation and analysis logbook pages,
  - Internal sample and sample extract transfer chain-of-custody records,
  - Screening records, and
  - All instrument output, including strip charts from screening activities.
- 2.7.2.6 All other original SDG-specific documents in the possession of the Contractor including, but not limited to, the following documents:
  - Telephone contact logs,
  - Copies of personal logbook pages,
  - All hand-written SDG-specific notes, and
  - Any other SDG-specific documents not covered by the above.
- 2.7.3 If the Contractor does submit SDG-specific documents to the RSCC after submission of the CSF, the documents should be identified with unique accountable numbers, a revised Form DC-2 should be submitted, and the unique accountable numbers and the locations of the documents in the CSF should be recorded in the "Other Records" section on the revised Form DC-2. Alternatively, the Contractor may number the newly submitted SDG-specific documents to the RSCC as a new CSF and submit a new Form DC-2.
- 2.8 GC/MS Tapes. The Contractor shall adhere to the requirements in Exhibit E.
- 2.9 GC Tapes. The Contractor shall adhere to the requirements in Exhibit E.
- 2.10 Extracts. The Contractor shall preserve sample extracts at less than 4°C but not greater than 6°C in bottles/vials with Teflon-lined septa. Extract bottles/vials shall be labeled with EPA sample number, Case number and SDG number. The Contractor shall maintain a logbook of stored extracts, listing EPA sample numbers and associated Case and SDG numbers. The Contractor shall retain extracts for 365 days following submission of the reconciled complete sample data package. During that time, the Contractor shall submit extracts and associated logbook pages

within seven days following receipt of a written request from the Project Officer.

- 2.11 Results of Quarterly Blind (QB) Performance Evaluation (PE) Program. The Contractor shall tabulate analytical results for the QB PE sample analyses, including all requirements specified in Section 2.7 above.
- 2.12 Corrective Action Procedures. If a Contractor fails to adhere to the requirements detailed in this SOW, a Contractor may expect, but the Agency is not limited to the following actions: reduction of numbers of samples sent under this contract, suspension of sample shipment to the Contractor, data package audit, an on-site laboratory evaluation, remedial performance evaluation sample, and/or contract sanctions, such as a Cure Notice (see Exhibit E for additional details).

### 3.0 FORMS INSTRUCTIONS

- 3.1 Introduction. This section includes specific instructions for completing the data reporting forms required under this contract. Each of the forms is specific to a given fraction (volatile, semivolatile, pesticide/Aroclor, etc.) and, in some instances, specific to a given matrix (water, soil or waste) within each fraction. The Contractor shall submit only those forms pertaining to the fractions analyzed for a given sample(s). For instance, if a sample is scheduled for volatiles analysis only, the Contractor shall provide only forms for the volatile fraction. NOTE: There are two pages relating to the volatile, semivolatile, modified 524.2 volatile fractions for Forms I, VI, VII, VIII and also Form IX for semivolatile. Whenever a fraction is analyzed which contains multiple forms, **both** pages (-1 and -2) shall be submitted.
- 3.2 General Information. The Contractor shall report values on the hardcopy forms according to the individual form instructions in this section. For example, results for concentrations of volatile target compounds shall be reported to two significant figures if the value is greater than or equal to 10.
  - 3.2.1 When submitting data, the Contractor shall reproduce **all** characters that appear on the data reporting forms in Section 4. The format of the forms submitted shall be identical to that shown in the contract. No information may be added, deleted, or moved from its specified position without prior written approval of the EPA. The names of the various fields and compounds (e.g., "Lab Code," "Chloromethane") shall appear as they do on the forms in the contract, including the options specified in the form (e.g., "Matrix: (water/soil/waste)") shall appear, not just "Matrix". For items appearing on the **uncompleted** forms (Section 4), the use of uppercase and lowercase letters is optional.
  - 3.2.2 Alphabetical entries made on the forms by the Contractor shall be in ALL UPPERCASE letters (e.g., "LOW", not "Low" or "low").
- 3.3 Header Information.
  - 3.3.1 General Header Information. Five pieces of information are common to the header section of each data reporting form: lab name, contract, lab code, Case number and SDG number. This information shall be entered on every form and shall match on every form.
    - 3.3.1.1 Lab Name. The lab name shall be the name chosen by the Contractor to identify the laboratory. It shall not exceed 25 characters.
    - 3.3.1.2 Contract. Contract refers to the number of the EPA contract under which the analyses were performed. This lab name will be the name identified at the time a Contract is awarded and shall not be modified by the Contractor, except at the direction of EPA.
    - 3.3.1.3 Lab Code. The lab code is an alphabetical abbreviation of up to six letters, as assigned by EPA, to identify the laboratory and aid in data processing. This lab code will be assigned by EPA at

the time a contract is awarded, and shall not be modified by the Contractor, except at the direction of the EPA. If a change of name or ownership occurs at the laboratory, the lab code will remain the same until the Contractor is directed by EPA to use another lab code.

- 3.3.1.4 Case Number. The Case number is the EPA-assigned Case number associated with the sample. This number is reported on the Traffic Report.
- 3.3.1.5 SDG Number. The "SDG No." field is for the sample delivery group number. It is the EPA sample number of the first sample received in the SDG. When several samples are received together in the first SDG shipment, the SDG number shall be the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG.
- 3.3.2 Sample Number Information. Sample numbering appears either in the upper righthand corner of the form, or as the left column of a table summarizing data from a number of samples. When the EPA sample number is entered in the triple-spaced box in the upper righthand corner of Form I, Form IV, or Form X, it should be entered in the middle of the box.
- 3.3.2.1 The Contractor shall identify **all** samples, including dilutions and reanalyses, matrix spikes, matrix spike duplicates, blanks, and standards with an EPA sample number. For field samples, matrix spikes and matrix spike duplicates, the EPA sample number is the unique identifying number given in the Traffic Report that accompanied that sample. In order to facilitate data assessment, the Contractor shall use the following sample suffixes:
- |           |   |   |
|-----------|---|---|
| XXXXXX    | = | EPA sample number   |
| XXXXXMS   | = | Matrix spike sample   |
| XXXXXXMSD | = | Matrix spike duplicate sample   |
| XXXXXXRE  | = | Re-extracted and reanalyzed sample  |
| XXXXXXDL  | = | The suffix DL is appended to the EPA sample number to indicate that the analytical results are a result of a dilution of the original analysis (reported as EPA sample XXXXXX). See Exhibit D for requirements for dilutions. |
- 3.3.2.2 There may be instances when all samples analyzed must be listed on the form, regardless of whether or not they are part of the SDG being reported (e.g., Form IX PEST). In these instances, use ZZZZZ as the EPA sample number for any sample analysis **not** associated with the SDG being reported.

Exhibit B--Section 3  
Forms Instructions

3.3.2.3 The EPA sample number to identify blanks shall use the following identification scheme: F\*BLK##, where

F	= Fraction code:	V	= Volatiles
		S	= Semivolatiles
		P	= Pesticides/Aroclors
		5	= Modified 524.2 Volatiles
		A	= Aroclors (only)
		N	= Water Soluble Organics - NPD
		F	= Water Soluble Organics - FID
*	= Type of blank:	M	= Method Blank
		I	= Instrument Blank
		H	= Storage Blank

BLK = Blank

## = One or two characters, numbers, or combination of both to create a unique EPA sample number within an SDG. If a method blank is analyzed on multiple instruments, then an additional two-character suffix shall be added to make the blank EPA sample number unique.

3.3.2.4 Standards shall be identified as F++++\*##, where

F	= Fraction code (specified in Section 3.3.2.3).
+++	= Type of Standard      STD = Standard (Initial calibration) ICV = Initial Calibration Verification CCS = Continuing Calibration Standard
***	= Standard concentration (specified for each method in Exhibit D). In cases where the target compounds are at different concentrations within a standard mixture, the lowest concentration shall prevail as the number inserted for ***.
##	= One or two characters, numbers, or combinations of both to create a unique EPA sample number within an SDG.

3.3.2.5 The Contractor shall use the following scheme to identify pesticide/Aroclor standards:

<u>Name</u>	<u>EPA Sample Number</u>
Individual Mix A (low point)	INDAL##
Individual Mix A (mid-point)	INDAM##
Individual Mix A (high point)	INDAH##
Individual Mix B (low point)	INDBL##
Individual Mix B (mid-point)	INDBM##
Individual Mix B (high point)	INDBH##
Resolution Check	RESC##
Performance Evaluation Mixture	PEM##
Toxaphene	TOXAPH##
Aroclor 1016	AR1016##
Aroclor 1221	AR1221##
Aroclor 1232	AR1232##
Aroclor 1242	AR1242##
Aroclor 1248	AR1248##
Aroclor 1254	AR1254##
Aroclor 1260	AR1260##
Aroclor 1016/1260 Mix	AR1660##
Aroclor 1262	AR1262##



<u>Name</u>	<u>EPA Sample Number</u>
Aroclor 1268	AR1268##

The Contractor shall replace the two-character terminator (##) of the identifier with one or two characters or numbers, or a combination of both, to create a unique EPA sample number within an SDG.

3.3.2.7 For all GC analyses, if the standards are injected onto both GC columns on the same instrument simultaneously, the same EPA sample number may be used for reporting data for the standards for both columns. If simultaneous injections are **not** made, then the same number shall **not** be used.

3.3.2.8 The EPA sample number for GPC shall be GPC#####, where ##### is the GPC column ID.

3.3.2.9 The EPA sample number for florisil shall be FLO#####, where ##### is the florisil cartridge lot number.

3.3.3 Other Common Fields. Several other pieces of information are common to many of the data reporting forms. These include matrix, sample weight/volume, level, lab sample identifier, and lab file identifier.

- In the "Matrix" field, enter SOIL for soil/sediment/solid samples, WATER for aqueous samples and WASTE for oily sludge/waste samples.

NOTE: The matrix shall be spelled out. Abbreviations such as S or W shall **not** be used.

- In the "Sample wt/vol" field, enter in the first blank the number of grams (for soil/sediment/solid or waste) or milliliters (for water) of sample used. Enter the units, either G or ML, in the second blank.
- The "Level" field is used for the volatile and semivolatile, fractions. Enter the determination of concentration level made from the screening of soils. Enter as LOW or MED, **not** L or M. All water samples shall be entered as LOW.

NOTE: There is no differentiation between low and medium soil samples for the remaining fractions, and no level is entered on any of these forms.

- The "Lab Sample ID" field is a unique laboratory-generated internal identifier pertaining to a particular sample. The Contractor may use the EPA sample number as the lab sample identifier.
- The "Lab File ID" field is the unique laboratory-generated name of the GC or GC/MS data system file containing information pertaining to a particular sample analysis.

3.3.3.1 The "Instrument ID" field is common to the forms containing calibration data. The identifier used by the Contractor shall include some indication of the manufacturer and/or model of the instrument, and shall contain additional characters that differentiate between all instruments of the same type in the laboratory.

3.3.3.2 Forms II, IV, V, IX, X and XI contain a field labeled "page \_\_\_ of \_\_\_" in the bottom lefthand corner. If the number of entries required on any of these forms exceeds the available space, continue entries on another copy of the same fraction-specific form, duplicating all header information. If a second page is required, number the pages consecutively (i.e., "page 1 of 2" and "page 2 of 2"). If a second page is **not** required, number the page "page 1 of 1."

NOTE: These forms are fraction-specific, and often matrix-specific within a fraction. For example, Form II VOA-1 and Form

II VOA-2 are for different data. Therefore, **do not** number the pages of all six versions of Form II as "1 of 6," "2 of 6," etc. Number only pages corresponding to the fraction-specific and matrix-specific form.

- 3.3.4 Rounding Rule. For rounding off numbers to the appropriate level of precision, the Contractor shall follow these rules. If the figure following those to be retained is less than 5, drop it (round down). If the figure is greater than 5, drop it and increase the last digit to be retained by 1 (round up). If the figure following the last digit to be retained equals 5, round up if the digit to be retained is odd, and round down if that digit is even.

3.4 Organic Analysis Data Sheet (Form I, All Fractions)

- 3.4.1 Purpose. This form is used for tabulating and reporting sample results for target compounds, including blanks, matrix spikes, and matrix spike duplicates. If all fractions are not requested for analysis, only the pages for the fractions required shall be submitted. For example, if only volatiles analysis is requested, Form I VOA-1, Form I VOA-2, and Form I VOA-TIC shall be submitted. If only the pesticide/Aroclor fraction is requested for analysis, Form I PEST shall be submitted. Furthermore, instrument blanks for dual column GC analyses shall be reported on a per column/per analysis basis on Form I. Each instrument blank shall be named with a unique EPA sample number.

- 3.4.2 Instructions. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

- 3.4.2.1 For soil samples analyzed for volatiles and water soluble organics (both GC/NPD and GC/FID) enter the non-decanted percent moisture in the "% Moisture: (not decanted)." field on the Form I pages of each analysis. This is the only percent moisture determination made for these volatile analyses since the entire contents of the VOA vial, or sample container, are considered as the sample. For water samples, leave this field blank.

- 3.4.2.2 For soil samples analyzed for the remaining fractions, enter the values for the percent moisture determined during the analysis in the "% Moisture" field on Form I. In the "decanted (Y/N)" field, enter Y if the sample had greater than 70 % moisture and the standing water above the soil/sediment/solid was decanted, or N if the sample had less than 70 % moisture and no water was decanted off the surface of the sample. Report percent moisture (decanted or not decanted) to the nearest whole percentage point (e.g., 5%, not 5.3%). For water samples and all required blanks leave these fields on Form I blank.

- 3.4.2.3 On Form I for volatiles and modified 524.2 volatiles, enter the GC column identifier in the "GC Column" field and in the "ID" field enter the length of the column in meters (m), the internal diameter in millimeters (mm), and the film thickness in micrometers (µm).

- 3.4.2.4 For pesticides/Aroclors and Aroclors only, enter the method of extraction in the "Extraction" field on Form I as SEPF for separatory funnel, CONT for continuous liquid-liquid extraction, or SONC for sonication (soils only).

- 3.4.2.5 If gel permeation chromatography (GPC) was performed, enter Y in the "GPC Cleanup" field on Form I for semivolatiles and pesticide/Aroclors. Enter N in this field if GPC was not performed.

NOTE: GPC is **required** for all **soil** samples analyzed for semivolatiles and pesticides/Aroclors; therefore, all forms for soil samples will contain a Y in this field.

- 3.4.2.6 For soil samples only, for semivolatiles, pesticides/Aroclors, and Aroclors only, enter the pH reported to 0.1 pH units, on their respective Form I pages.

- 3.4.2.7 Enter the date of sample receipt at the laboratory, as noted on the Traffic Report (i.e., the VTSR), in the "Date Received" field. The date shall be entered as MM/DD/YY.
- 3.4.2.8 Complete the "Date Extracted" and "Date Analyzed" fields in the same format (MM/DD/YY). When continuous liquid-liquid extraction procedures are used for water samples, enter the date that the procedure was **started** in the "Date Extracted" field. If separatory funnel or sonication procedures are used, enter the date that the procedure was **completed** in the "Date Extracted" field. The date of sample receipt will be compared with the extraction and analysis dates of each fraction to ensure that contract holding times were not exceeded.
- 3.4.2.9 If a medium soil/sediment/solid or waste sample is analyzed for volatiles, enter total volume of the methanol extract in microliters (uL) in the "Soil Extract Volume" field on their respective Form I pages. This volume includes any methanol not collected from the filtration of the extract through glass wool; the volume is typically 10,000 uL (i.e., the 10 mL of methanol used for the extraction). If a medium soil sample is analyzed, enter the volume of the methanol extract added to the reagent water in the purge tube and analyzed in the "Soil Aliquot Volume" field. Enter this volume in microliters (uL).
- 3.4.2.10 For semivolatiles, pesticides/Aroclors, and Aroclors only, enter the actual volume of the **most** concentrated sample extract, in microliters (uL), in the "Concentrated Extract Volume" field on their respective Form I pages. For semivolatiles, this volume will typically be 1,000 uL (for water) or 500 uL (for water and soil) when GPC is performed. For pesticides/Aroclors, the volume of the most concentrated extract will typically be 10,000 uL (for water) or 5,000 uL (for water and soil) when GPC is performed. For Aroclors only, the volume of the most concentrated extract will typically be 10,000 uL (for water and soil). For pesticides/Aroclors and Aroclors only the volume of the most concentrated extract is **not** the volume taken through the Florisil, sulfur cleanup or acid cleanup steps. If a dilution of the sample extract is made in a subsequent analysis, this volume will remain the same, but the dilution factor will change.
- 3.4.2.11 For water soluble organics (GC/NPD and GC/FID), for soil/sediment/solid samples, enter the actual volume of water, in milliliters, added to the sample to perform the extraction in the "Soil Extraction Volume" field. This volume is typically 40 mL.
- 3.4.2.12 For semivolatiles, pesticides/Aroclors, Aroclors only, and water soluble organics (GC/NPD and GC/FID), enter the volume of the sample or sample extract injected into the GC in the "Injection Volume" field on their respective Form I pages. Report this volume in microliters (uL) to one decimal place (e.g., 1.0 uL).
- NOTE: A 2.0 microliter injection is **required** for semivolatile analyses.
- 3.4.2.13 For dual column/single injector GC analyses, enter the amount of half the volume in the syringe in the "Injection Volume" field (i.e., assume that the extract injected is evenly divided between the two columns).
- 3.4.2.14 If a sample or sample extract has been diluted for analysis, enter the dilution factor as a single number (e.g., enter 100.0 for a 1 to 100 dilution of the sample) in the "Dilution Factor" field. The dilution factor shall not be entered as a fraction. If a sample was not diluted, enter 1.0. Report dilution factors to one decimal place.
- 3.4.2.15 If sulfur cleanup is employed for pesticide/Aroclors or Aroclors only analyses, enter Y in the "Sulfur Cleanup" field; if not, enter N on Form I PEST or Form I PCB, respectively.
- 3.4.2.16 If sulfuric acid cleanup is employed for the analysis of Aroclors in pesticide/Aroclors or Aroclors only analyses, enter Y in the

"Aroclor Acid Cleanup" field. All Aroclors only analyses require sulfuric acid cleanup.

- 3.4.2.17 For positively identified target compounds, the Contractor shall report the concentrations as **uncorrected** for blank contaminants.
- 3.4.2.18 Report all analytical results to one significant figure if the value is less than 10, and two significant figures if the value is 10 or above. Report all modified 524.2 volatile and pesticide/Aroclor results to two significant figures.
- 3.4.2.19 Enter the appropriate concentration units, ug/L or ug/Kg. (Note: Water soluble organics - GC/FID results are reported in mg/L and mg/Kg.)
- 3.4.2.20 Under the column labeled "Q" for qualifier, flag each result with the specific data reporting qualifiers listed below. When reporting results to EPA, the Contractor shall use these contract-specific qualifiers. The Contractor shall not modify the qualifiers. Up to five qualifiers may be reported on Form I for each compound. The Contractor is encouraged to use additional flags or footnotes (see the X qualifier).

The EPA-defined qualifiers to be used are:

U: This flag indicates the compound was analyzed for but not detected. The CRQL shall be adjusted according to the equation listed in Exhibit D. CRQLs are listed in Exhibit C.

J: This flag indicates an estimated value. This flag is used (1) when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed, (2) when the mass spectral and retention time data indicate the presence of a compound that meets the volatile, modified 524.2 volatile and semivolatile GC/MS identification criteria, and the result is less than the CRQL but greater than zero, and (3) when the retention time data indicate the presence of a compound that meets the identification criteria, and the result is less than the CRQL but greater than zero. For example, if the sample quantitation limit is 10 ug/L, but a concentration of 3 ug/L is calculated, report it as 3J.

NOTE: The J flag is not used and the compound is not reported as being identified for results less than the CRQL if the GC chromatography expert determines that the peaks used for compound identification resulted from instrument noise or other interferences (column bleed, solvent contamination, etc.).

N: This flag indicates presumptive evidence of a compound. This flag is only used for tentatively identified compounds (TICs), where the identification is based on a mass spectral library search. It is applied to all TIC results. For generic characterization of a TIC, such as chlorinated hydrocarbon, the N flag is not used.

P: This flag is used for a target analyte when there is greater than 25% difference for detected concentrations between the two GC columns (see Form X). **The lower of the two values is reported on Form I and flagged with a P.**

C: This flag applies to GC results where the **identification** has been confirmed by GC/MS. If GC/MS confirmation was attempted but was unsuccessful, do **not** apply this flag; use a laboratory-defined flag instead (see the X qualifier).

B: This flag is used when the analyte is found in the associated method blank as well as in the sample. It indicates probable blank contamination and warns the data user to take appropriate action. This flag shall be used for a

tentatively identified compound as well as for a positively identified target compound.

The combination of flags BU or UB is expressly prohibited. Blank contaminants are flagged B only when they are detected in the sample.

- E: This flag identifies compounds whose concentrations exceed the upper level of the calibration range of the instrument for that specific analysis. If one or more compounds have a response greater than the upper level of the calibration range, the sample or extract shall be diluted and reanalyzed according to the specifications in Exhibit D; exceptions are also noted in Exhibit D. All such compounds with a response greater than the upper level of the calibration range shall have the concentration flagged with an E on Form I for the original analysis.

NOTE: For total xylenes, where three isomers are quantified as two peaks, the calibration range of **each peak** shall be considered separately. For example, a diluted analysis is **not** required for total xylenes unless the concentration of the peak representing the single isomer exceeds 200 ug/L or the peak representing the two co-eluting isomers on that GC column exceeds 400 ug/L.

- D: If a sample or extract is reanalyzed at a higher dilution factor, for example when the concentration of an analyte exceeds the upper calibration range, the DL suffix is appended to the sample number on Form I for the more diluted sample, and **all** reported concentrations on that Form I are flagged with the D flag. This flag alerts data users that any discrepancies between the reported concentrations may be due to dilution of the sample or extract. NOTE 1: The D flag is not applied to compounds which are not detected in the sample analysis i.e. compounds reported with the CRQL and the U flag. NOTE 2: Separate Form Is are required for reporting the original analysis (EPA Sample No. XXXXX) and the more diluted sample analysis (EPA Sample No. XXXXXDL) i.e. the results from both analyses cannot be combined on a single Form I.
- A: This flag indicates that a tentatively identified compound is a suspected aldol-condensation product.
- X: Other specific flags may be required to properly define the results. If used, the flags shall be fully described, with the description attached to the sample data summary package and the SDG Narrative. Begin by using X. If more than one flag is required, use Y and Z as needed. If more than five qualifiers are required for a sample result, use the X flag to represent a combination of several flags. For instance, the X flag might combine the A, B, and D flags for some samples. The laboratory-defined flags **are limited to X, Y, and Z.**

3.5 Organic Analysis Data Sheet: Tentatively Identified Compounds (Form I VOA-TIC, 524.2-TIC and SV-TIC)

- 3.5.1 Purpose. This form is used to report analysis results for non-target compounds (e.g., compounds not listed in Exhibit C), excluding system monitoring compounds, surrogates and internal standards. See Exhibit D for instructions on identification and quantitation. The Contractor shall submit Form I VOA-TIC, SV-TIC or 524.2-TIC for **every analysis**, including required dilutions and reanalyses, even if no TICs are found.
- 3.5.2 Instructions. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions in addition to the instructions in Section 3.4.

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Forms Instructions

- 3.5.2.1 Report all TICs including CAS number (if applicable), compound name, retention time, and the estimated concentration as uncorrected for blank contaminants. If the analytical result is less than 10, report to one significant figure. If the analytical result is 10 or greater, report to two significant figures. (Criteria for reporting TICs are given in Exhibit D, Section 11). Retention time shall be reported in minutes and decimal minutes, **not** seconds or minutes:seconds. If, in the opinion of the mass spectral interpretation specialist, no valid tentative identification can be made, the compound shall be reported as unknown.
- 3.5.2.2 Total the number of TICs found, **including** aldol-condensation products (see Section 3.5.2.3), and enter this number in the "Number TICs found" field. If no TICs were found, enter 0 (zero).
- 3.5.2.3 Peaks that are suspected to be aldol-condensation reaction products (e.g., 4-methyl-4-hydroxy-2-pentanone and 4-methyl-3-pentene-2-one) shall be summarized on this form as additional compounds, flagged A, and included in the "Number TICs found" field. The peaks shall be counted as additional TICs to the 10 most intense non-target compounds to be searched.
- 3.6 System Monitoring Compound Recovery (Form II VOA and Form II 524.2)
- 3.6.1 Purpose. For volatiles and modified 524.2 volatiles, Form II is used to report the recoveries of the system monitoring compounds added to each volatile sample, including each dilution and reanalysis, blank, matrix spike, and matrix spike duplicate. The system monitoring compounds are used to monitor the performance of the purge and trap GC/MS system as a whole. Form II VOA is matrix-specific, so that system monitoring compound recoveries for water samples are reported on a different version of Form II than the recoveries for soil/sediment/solid samples. The matrix specific form for volatile soil samples shall be used for both soil/sediment/solid samples and oily sludge/waste samples. In the "Matrix (soil/waste)" field, enter SOIL for soil/sediment/solid samples and WASTE for oily sludge/waste samples. A separate form must be used for each matrix type. Soil/sediment/solid sample recoveries are further differentiated by concentration level.
- 3.6.2 Instructions. Complete the header information according to the instructions in Section 3.3. NOTE: For volatile **soil** samples only, specify the level as LOW or MED. Complete one form for each level. **Do not** mix low and medium level samples on one form. Complete the remainder of the form using the following instructions.
- 3.6.2.1 For each system monitoring compound listed in Table 3, report the percent recovery to the nearest whole percentage point, and to the number of significant figures given by the QC limits at the bottom of the form.
- 3.6.2.2 Flag each system monitoring compound recovery outside the QC limits with an asterisk (\*). The asterisk shall be placed in the last space in each appropriate column, under the "#" symbol.
- 3.6.2.3 In the "TOT OUT" column, total the number of system monitoring compound recoveries that were outside the QC limits for each sample. If no system monitoring compounds were outside the limits, enter 0 (zero).
- 3.6.2.4 Number all pages as described in Section 3.3.

**Table 3**  
**System Monitoring Compounds**

<b>Volatile System Monitoring Compounds</b>	<b>CAS Number</b>
SMC 1: 1,2-Dichloroethane-d4 (DCE)	17060-07-0

SMC 2: 1,2-Dichlorobenzene-d4 (DCB) 2199-69-1

Low Concentration Volatile System Monitoring Compounds	CAS Number
---	------------

SMC 1: 1,2-Dichloroethane-d4 (DCE)	17060-07-0
------------------------------------	------------

SMC 2: 1,2-Dichlorobenzene-d4 (DCB)	2199-69-1
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### 3.7 Surrogate Recovery (Form II SV, PEST, PCB, WSO/NPD, WSO/FID)

3.7.1 Purpose. Form II is used to report the recoveries of the surrogate compounds added to each sample, blank, duplicate, matrix spike, and matrix spike duplicate. Form II is matrix-specific as well as fraction-specific, so surrogate recoveries for water samples are reported on a different version of Form II than surrogate recoveries for soil/sediment/solid samples. The matrix specific form for soil samples shall be used for both soil/sediment/solid samples and oily sludge/waste samples. In the "Matrix (soil/waste)" field, enter SOIL for soil/sediment/solid samples and WASTE for oily sludge/waste samples. A separate form must be used for each matrix type.

3.7.2 Instructions. Complete the header information according to the instructions in Section 3.3. NOTE: For **semivolatile soil** samples only, specify the level as LOW or MED. Complete one form for each level. **Do not** mix low and medium level samples on one form. Complete the remainder of the form using the following instructions.

3.7.2.1 For each surrogate listed in Table 4, report the percent recovery to the nearest whole percentage point.

3.7.2.2 Flag each surrogate recovery outside the QC limits with an asterisk (\*). The asterisk shall be placed in the last space in each appropriate column, under the "#" symbol.

3.7.2.3 In the "TOT OUT" column, total the number of surrogate recoveries that were outside the QC limits for each sample. If no surrogates were outside the limits, enter 0 (zero).

3.7.2.4 If the sample is diluted and the surrogates are outside the acceptance window in any analysis, enter the calculated recovery, and flag the surrogate recoveries with a D in the column under the "#" symbol. **Do not** include results flagged with a D in the total number of recoveries for each sample outside the QC limits.

3.7.2.5 Surrogate recoveries for dual column analyses shall be reported from **both** GC columns used. Therefore, identify each GC column at the top of Form II, entering the column identifier in the "GC Column" field, and in the "ID" field enter the length of the column in meters (m), the internal diameter in millimeters (mm) and the film thickness in micrometers (µm).

3.7.2.6 The assignment of columns as "1" and "2" is left to the discretion of the Contractor when the analyses are performed by simultaneous injection into a GC containing two columns. If so analyzed, the assignment of "GC Column 1" and "GC Column 2" shall be consistent across all the reporting forms. If the analysis is **not** performed by simultaneous injection, then the assignment of GC column number shall be based on the chronological order of the two analyses.

3.7.2.7 Although surrogate recovery limits for samples, matrix spike and matrix spike duplicates are only advisory, the Contractor shall flag those recoveries that are outside the advisory QC limits or are diluted out. The total number of recoveries that are outside the QC limits shall include all values from both GC columns. In counting the total number of recoveries that are outside the QC limits, do not include the results flagged with a D.

3.7.2.8 Number all pages as described in Section 3.3.

**Table 4**  
**Surrogate Compounds**

<b>Semivolatile Surrogates</b>	<b>CAS Number</b>
S1: Nitrobenzene-d5 (NBZ)	4165-60-0
S2: 2-Fluorobiphenyl (FBP)	321-60-8
S3: Terphenyl-d14 (TPH)	98904-43-9
S4: Phenol-d5 (PHL)	4165-62-2
S5: 2-Fluorophenol (2FP)	367-12-4
S6: 2,4,6-Tribromophenol (TBP)	118-79-6
S7: 2-Chlorophenol-d4 (2CP)	93951-73-6
S8: 1,2-Dichlorobenzene-d4 (DCB)	2199-69-1

  

<b>Pesticide/Arochlor and Aroclor only Surrogates</b>	<b>CAS Number</b>
Decachlorobiphenyl (DCB)	2051-24-3
Tetrachloro-m-xylene (TCX)	877-09-8

  

<b>Water Soluble Organics Surrogates</b>	<b>CAS Number</b>
NPD - N,N-Dimethylacetamide (DMA)	127-19-5
FID - 2-Ethoxyethanol (2EE)	110-80-5

3.8 Matrix Spike/Matrix Spike Duplicate Recovery (Form III, All Fractions)

- 3.8.1 Purpose. This form is used to report the results of the analyses of matrix spikes and matrix spike duplicates (MS/MSD). The form is matrix-specific for all fractions. The matrix specific form for soil samples shall be used for both soil/sediment/solid samples and oily sludge/waste samples. In the "Matrix (soil/waste)" field, enter SOIL for soil/sediment/solid samples and WASTE for oily sludge/waste samples. A separate form must be used for each matrix type.
- 3.8.2 Instructions. Complete the header information according to the instructions in Section 3.3. Include the EPA sample number for the matrix spike, **without** the suffixes MS or MSD. Complete the remainder of the form using the following instructions.
- 3.8.2.1 For volatile and semivolatile soil samples, specify level as LOW or MED on Form III VOA or SV. SDGs containing soil samples at both levels require a MS/MSD at each level; therefore, for soils, prepare one form for each level.
- 3.8.2.2 In the first table under the "SPIKE ADDED" column, enter the calculated concentration in ug/L or ug/Kg (according to the matrix) that results from dividing each spike compound amount added to the aliquot weight/volume chosen for the matrix spike. For instance, for base/neutral compounds in medium level soils, if 50 ug of spike are added to 1 g of soil, the resulting concentration is 50,000 ug/Kg.
- 3.8.2.3 Enter the sample concentration in the next column, in similar units, of each spike compound detected in the original sample. If a spike compound was not detected during the analysis of the original sample, enter the sample result as 0 (zero).
- 3.8.2.4 In the "MS CONCENTRATION" column, enter the actual concentration of each spike compound detected in the matrix spike aliquot.



- 3.8.2.5 Calculate the percent recovery of each spike compound in the matrix spike aliquot to the nearest whole percent, according to Exhibit D. Enter the percent recovery in the "MS % REC" column.
- 3.8.2.6 Flag all percent recoveries outside the QC limits with an asterisk (\*). The asterisk shall be placed in the last space of the "MS % REC" column, under the "#" symbol.
- 3.8.2.7 For dual column analyses, the MS and the MSD concentrations shall be the concentration of the spiked analyte reported on Form I for those analyses. Of the two concentrations calculated for each target compound, one on each GC column, the **lower** concentration shall be reported on Form I, and **both** concentrations shall be reported on Form XI. The **lower** concentration is also reported on Form III and used in the calculation of spike recovery, even if that concentration yields a recovery value that is outside the advisory QC limits.
- 3.8.2.8 Follow Sections 3.8.2.2 through 3.8.2.7 to complete the lower table, using the results of the analysis of the MSD aliquot.
- 3.8.2.9 Calculate the relative percent difference (RPD) between the matrix spike recovery and the matrix spike duplicate recovery, and enter this value in the "% RPD" column. Report the RPD to the nearest whole percent.
- 3.8.2.10 Compare the RPDs to the QC limits given on the form, and flag each RPD outside the QC limits with an asterisk (\*) in the last space of the "% RPD" column, under the "#" symbol.
- 3.8.2.11 Summarize the values outside the QC limits at the bottom of the page. No further action is required by the Contractor.
- 3.9 Method Blank Summary (Form IV, All Fractions)
- 3.9.1 Purpose. This form summarizes the samples associated with each method blank analysis. The Contractor shall submit the appropriate Form IV for each blank.
- 3.9.2 Instructions. Complete the header information according to the instructions in Section 3.3. The EPA sample number entered in the upper righthand corner shall be the same number entered on Form I for the blank. Complete the remainder of the form using the following instructions.
- 3.9.2.1 Complete the following fields: "Instrument ID," "Date Analyzed," and "Time Analyzed." Dates shall be entered as MM/DD/YY. The time shall be reported in military time in hours, minutes and decimal minutes.
- 3.9.2.2 For dual column analyses, contaminants shall meet the identification criteria requiring analysis of the blank on two different GC columns. Enter the date, time and instrument ID of both blank analyses on the method blank summary (Form IV). The information on the two analyses is differentiated as Date Analyzed (1), Date Analyzed (2), etc. If the analyses were run simultaneously, the order of reporting is not important, but shall be consistent with the information reported on all other forms for that fraction. Otherwise, Date Analyzed (1) shall indicate the analysis on column 1, and Date Analyzed (2) shall indicate the analysis on column 2.
- 3.9.2.3 Identify the GC column, including the length, internal diameter and film thickness in the appropriate fields.
- 3.9.2.4 For volatiles, indicate the purging method by entering Y for heated purge or N for ambient temperature purge in the "Heated Purge: Y/N" field on Form IV VOA.
- 3.9.2.5 For pesticide/Aroclor and Aroclor only, enter the method of extraction as SEPF for separatory funnel, SONC for sonication, or CONT for continuous liquid-liquid extraction on Form IV PEST or Form IV PCB.

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- 3.9.2.6 For method blanks involving an extraction process, record the date of extraction on Form IV.
- 3.9.2.7 If the samples associated with the pesticide/Aroclor or Aroclor only blank are subjected to sulfur cleanup, then the blank shall also be subjected to sulfur cleanup. If sulfur cleanup is employed, enter Y in the "Sulfur Cleanup" field; if not, enter N on Form IV PEST or Form IV PCB. If only some of the samples associated with the method blank are subjected to sulfur cleanup, a **separate** sulfur cleanup blank is required (see Exhibit D). If a separate sulfur cleanup blank is prepared, complete one version of Form IV associating all the samples with the method blank, and a second version of Form IV listing only those samples associated with the separate sulfur cleanup blank. NOTE: Subjecting all samples associated with a method blank to sulfur cleanup avoids the need for two forms.
- 3.9.2.8 If the samples associated with the pesticide/Aroclor blank are subjected to a sulfuric acid cleanup, then a portion of the blank shall also be subjected to the sulfuric acid cleanup. If sulfuric acid cleanup is performed on portion of the Pesticide/Aroclor extract for the determination Aroclors, create a separate Form IV, change the last 2-digit numbering code of the EPA Sample No. to differentiate between the method blanks, and enter Y in the "Aroclor Acid Cleanup" field on Form IV PEST. If a separate acid cleanup blank is prepared, complete one version of Form IV associating all the samples with the method blank, and a second version of Form IV listing only those samples associated with the separate sulfuric acid cleanup blank.
- 3.9.2.9 For all fractions, as appropriate, summarize the samples, QC sample and required blanks, associated with a given method blank in the table, entering the EPA sample number and lab sample identifier. For volatiles and modified 524.2 volatiles, enter the lab file identifier and the time of analysis of each sample. For semivolatiles, enter lab file identifier and the date of analysis. For all dual column analyses, enter the dates of both analyses as Date Analyzed (1) and Date Analyzed (2), as discussed previously.
- 3.9.2.10 Number all pages as described in Section 3.3.
- 3.10 GC/MS Instrument Performance Check and Mass Calibration (Form V VOA, SV and 524.2)
- 3.10.1 Purpose. This form is used to report the results of the GC/MS instrument performance check for the volatile, modified 524.2 volatile and semivolatile fractions and to summarize the date and time of analyses of samples, including dilutions and reanalyses, standards, blanks, matrix spikes, and matrix spike duplicates associated with each analysis of the instrument performance check solution.
- 3.10.2 Instructions. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.
- 3.10.2.1 Enter the date and time of injection of the instrument performance check solution (BFB for volatiles and modified 524.2 volatiles, CAS Number 460-00-4, and DFTPP for semivolatiles, CAS Number 5074-71-5). The date shall be entered as MM/DD/YY. The time shall be reported as military time in hours, minutes and decimal minutes.
- 3.10.2.2 For volatiles and modified 524.2 volatiles, identify the GC column, including the length, internal diameter and film thickness on Form V.
- 3.10.2.3 For volatiles, indicate the purging method by entering Y for heated purge or N for ambient temperature purge in the "Heated Purge: Y/N" field on Form V. Water samples and medium soil sample extracts may be reported on the same Form V if analyzed together, since a single calibration may be applied to both sample types.

- 3.10.2.4 For each ion listed on the form, enter the percent relative abundance in the righthand column of the first table. Report relative abundances to the number of significant figures given for each ion in the ion abundance criteria column.
- NOTE: For both BFB and DF TPP, one or more of the high mass ions may exceed the abundance of the ion listed on the form as the nominal base peak, m/z 95 for BFB and m/z 198 for DF TPP. Despite this possibility, all ion abundances shall be normalized to the nominal base peaks listed on Form V (see Exhibits D and E).
- 3.10.2.5 All relative abundances shall be reported as a number. If the relative abundance is zero, enter 0, not a dash or other non-numeric character. Where parentheses appear, compute the percentage of the ion abundance of the mass given in the appropriate footnote, and enter that value in the parentheses.
- 3.10.2.6 In the lower table, list all samples, including dilutions and reanalyses, standards, blanks, matrix spikes, and matrix spike duplicates analyzed under that instrument performance check in chronological order, by time of analysis (in military time). Refer to Section 3.3.7 for specific instructions for identifying standards and blanks.
- 3.10.2.7 Complete the following fields for all standards, samples, including dilutions and reanalyses, blanks, matrix spikes, and matrix spike duplicates: "EPA Sample No.," "Lab Sample ID," "Lab File ID," "Date Analyzed," and "Time Analyzed."
- 3.10.2.8 Number all pages as described in Section 3.3.

3.11 GC/MS Initial Calibration Data (Form VI VOA, SV and 524.2)

- 3.11.1 Purpose. After a GC/MS system has undergone an initial five-point<sup>1</sup> calibration at the specific concentration levels described in Exhibit D, and after all initial calibration criteria have been met, the Contractor shall complete and submit this form for each volatile, modified 524.2 volatile or semivolatile target compound initial calibration performed which is relevant to the samples, including dilutions and reanalyses, blanks, matrix spikes, or matrix spike duplicates in the SDG, regardless of when that calibration was performed.
- 3.11.2 Instructions. Complete the header information according to the instructions in Section 3.3. Enter the Case number and SDG number for the current data package, regardless of the original Case for which the initial calibration was performed. Complete the remainder of the form using the following instructions.
- 3.11.2.1 Enter the date(s) of the calibration. If the calendar date changes during the calibration procedure, the inclusive dates shall be recorded. Dates shall be entered as MM/DD/YY.
- 3.11.2.2 Enter the injection times of the first and last of the standards analyzed in the "Calibration Times" field. Times shall be reported in military time in hours, minutes, and decimal minutes.
- 3.11.2.3 For volatiles and modified 524.2 volatiles, complete the "GC Column," and "ID" fields as on Form V, and for volatiles complete the "Heated Purge" field.
- 3.11.2.4 Enter the lab file identifier for each of the five calibration standards injected. Complete the response factor data for the five calibration points, and then calculate and report the average relative response factor (RRF) for all target compounds.
- 3.11.2.5 For volatiles and modified 524.2 volatiles, report the relative response factors for the system monitoring compounds in the calibration standards. For semivolatiles, report the response factors for all surrogate compounds in the calibration standards. The Contractor shall report the relative standard deviation (%RSD) for **all** compounds. See Exhibit D for equations.

3.12 GC/EC Pesticide/Aroclor Initial Calibration Data (Form VI PEST)

- 3.12.1 Purpose. The initial calibration of pesticides/Aroclors involves the determination of retention times, retention time windows, and calibration factors. For single component pesticide target compounds, these data are calculated from the analysis of the Individual Standard Mixtures A and B at three different concentration levels. For multicomponent target compounds, these data are calculated from a single point calibration.
- 3.12.2 Instructions. Complete one Form VI for **each** GC column used for the three analyses of Individual Standard Mixture A (low point, mid-point, and high point) and the three analyses of Individual Standard Mixture B during an initial calibration. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.
- 3.12.2.1 Enter the date of analysis of the standards on each form in the "Date(s) Analyzed" field. If the analysis of the standards carries over into the following day, list both the start and completion dates. Dates shall be entered as MM/DD/YY.

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<sup>1</sup>For semivolatiles, eight compounds (2,4-Dinitrophenol, 2,4,5-Trichlorophenol, 2-Nitroaniline, 3-Nitroaniline, 4-Nitroaniline, 4-Nitrophenol, 4,6-Dinitro-2-Methylphenol, and Pentachlorophenol) will only require a four-point initial calibration at 50, 80, 120, and 160 total nanograms because detection at less than 50 ng per injection is difficult. If a four-point calibration is performed for these compounds, leave the "RRF20" column blank.

- 3.12.2.2 The "Level (x low)" field shall be completed by entering the concentration of the low point, mid-point, and high point calibration standards as a multiplier of the low point. Thus, the low point will always be entered as "1.0". The concentrations of the mid-point and high point standards are specified in Exhibit D for each analytical method. The high point standard shall be at least 12 times the low point, but may be higher, and the value must lie within the linear range of the instrument. The appropriate mid and high point multipliers must be determined to one decimal place.
- 3.12.2.3 Using the same assignment of first and second GC columns made for Form IV, identify the GC column number and name, including the length (m), internal diameter (mm) and film thickness (μm) in the appropriate fields.
- 3.12.2.4 For each standard analyzed, enter the retention time of each applicable analyte in minutes and decimal minutes, under the appropriate concentration level in the "RT OF STANDARDS" column on Form VI PEST-1.
- 3.12.2.5 Calculate the mean retention time of each analyte from the three individual mixtures, and report it in the "MEAN RT" column on Form VI PEST-1.
- 3.12.2.6 Calculate the retention time window for each analyte using the specifications in Exhibit D, and enter the lower limit of the window in the "RT WINDOW" column under "FROM," and the upper limit of the window under "TO" on Form VI PEST-1. The retention times of the surrogates are reported from the analyses of Individual Mixture A and the windows are only required to be calculated for Individual Mixture A.
- 3.12.2.7 For the six analyses of the Individual Standard Mixtures, the Contractor shall also complete the calibration factor data on Form VI PEST-2. Prepare one form for each instrument and GC column used. Enter the calibration factor for each compound in each of the standards. Calculate and enter a mean calibration factor and the percent relative standard deviation (%RSD). As with surrogate retention times, the surrogate calibration factors are only required from Individual Mixture A analyses.
- 3.12.2.8 For the multicomponent target compounds, the retention times, retention time windows, and calibration factors shall be reported in a similar fashion for each single point calibration standard. For each multicomponent compound, the Contractor shall select at least three peaks from each analyte, according to the specifications in Exhibit D. The retention time and calibration factor data apply to **each** peak. Complete one version of Form VI PEST-3, PEST-4 for each GC column, for each initial calibration that applies to samples in the data package.
- 3.12.3 Form VI is also used to report the results of analysis of the Resolution Check Solution that shall begin each pesticide/Aroclor initial calibration sequence (Form VI PEST-5). The Contractor shall submit one Form VI PEST-5 for **both** GC columns.
- 3.12.4 Complete the header information as described in Section 3.3. Using the same assignment of first and second GC columns made for Form IV, enter the GC column identifier, length (m), internal diameter (mm), film thickness (μm) and date and time of analysis. Enter the EPA sample number for the Resolution Check Standard. If simultaneous injections on a single GC are used, the EPA sample number may be the same for both Resolution Check Standards. If simultaneous injections are **not** used, use different suffixes to identify the standards. Complete the remainder of the form using the following instructions.
- 3.12.4.1 List each analyte, in **retention time order**, including both surrogate compounds. Thus, the order of analytes in the two boxes on this form will be different due to the dissimilarity of the stationary phases of the two GC columns used. Enter the name of each target analyte in the Resolution Check Mixture as it appears

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on Form I PEST. Spell out the names of the surrogates as they appear on Form VII PEST-2.

- 3.12.4.2 Enter the retention time of each analyte from the analysis in the "RT" column.
- 3.12.4.3 Calculate the resolution between each pair of analytes. Enter the resolution between the first and second peaks on the line for the first analyte listed in the box. Enter the resolution between the second and third peaks on the line for the second analyte, and so on, until the resolutions of all possible pairs of adjacent analytes have been entered. NOTE: Only eight of the nine resolution fields will be filled.
- 3.12.5 Form VI (PEST-6, PEST-7 and PEST-8 for each pair of PEM, mid-level initial calibration mixture A, and mid-level initial calibration mixture B, respectively) shall be used to report the percent resolution between each pair of analytes according to the definition in Exhibit D (Pesticides).
- 3.12.6 Complete the header information as described in Section 3.3. Enter the EPA sample number for the respective standards. If simultaneous injections are **not** used, use different suffixes to identify the standards. Using the same assignment of first and second GC columns made for Form IV, enter the GC column name and identify the column length (m), internal diameter (mm) and phase thickness ( $\mu\text{m}$ ). Enter the Date of analysis as MM/DD/YY and time of analysis in minutes and decimal minutes. Complete the remainder of the form using the following instructions.
  - 3.12.6.1 List each analyte, in **retention time order**, including both surrogate compounds. Thus, the order of analytes in the two boxes on this form will be different due to the dissimilarity of the stationary phases of the two GC columns used. Enter the name of each target analyte in the standard as it appears on Form I PEST. Spell out the names of the surrogates as they appear on Form VII PEST-2.
  - 3.12.6.2 Enter the retention time of each analyte from the analysis in the "RT" column.
  - 3.12.6.3 Calculate the resolution between each pair of analytes. Enter the resolution between the first and second peaks on the line for the first analyte listed in the box. Enter the resolution between the second and third peaks on the line for the second analyte, and so on, until the resolutions of all possible pairs of adjacent analytes have been entered. NOTE: The last resolution field will be left blank in each table.

3.13 GC/EC Aroclor only Initial Calibration (Form VI PCB)

- 3.13.1 Purpose. The initial calibration of Aroclors involves the determination of retention times, retention time windows, and calibration factors. For Aroclors that are present in any of the samples, this data shall be calculated from the analyses of individual Aroclor standards at three different concentration levels. For Aroclors not present in any of the samples being analyzed this data shall be calculated from a single point calibration. If the analysis of calibration standards is required following sample analysis (see Section 9.2, Initial Calibration in Exhibit D, Aroclors), prepare and submit in chronological an additional Form VI.
- 3.13.2 Instructions. Complete one Form VI for **each** GC column used during the initial calibration for each Aroclor standard analyzed. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.
- 3.13.2.1 Enter the date of analysis of the standards on each Form VI in the "Date(s) Analyzed" field. If the analysis of the standards carries over into the following day, list both the start and completion dates. Dates shall be entered as MM/DD/YY.
- 3.13.2.2 The "Level (x low)" field shall be completed by entering the concentration of the low point, mid-point, and high point calibration standards as a multiplier of the low point. Thus, the low point shall always be entered as "1.0". The concentrations of the mid-point and high point standards are specified in Exhibit D for each analytical method. The high point standard shall be at least 10 times the low point, but may be higher, and the value must lie within the linear range of the instrument. The appropriate mid and high point multipliers must be determined to one decimal place and entered in the "Level (x low)" field.
- 3.13.2.3 For each target compound and surrogate, enter the actual concentration of the low point standard under the "LOW STD CONC." column on Form VI PCB-2. For single point Aroclor calibrations, enter the low point concentration established in Exhibit D that shall be used if a three point calibration is employed.
- 3.13.2.4 Using the same assignment of first and second GC columns made for Form IV, identify the GC column number and name, including the length (m), internal diameter (mm) and film thickness (µm) in the appropriate fields.
- 3.13.2.5 For each Aroclor standard and surrogate analyzed, enter the retention times of the three to five peaks established according to the specifications in Exhibit D, in minutes and decimal minutes, under the "RT OF STANDARDS" column on Form VI PCB-1. Prepare one form for each instrument and GC column used. For single point Aroclor calibrations, enter the appropriate peak retention times for the mid point standard under the "MID" column and leave the LOW and HIGH fields blank.
- 3.13.2.6 For each individual Aroclor and surrogate analyzed at three concentration levels, calculate the mean retention time of each peak, and report it in the "MEAN RT" column on Form VI PCB-1. This column shall remain blank for those Aroclors calibrated using a single standard.
- 3.13.2.7 Calculate the retention time window for each Aroclor and surrogate peak using the specifications in Exhibit D. Enter the lower limit of the window in the "RT WINDOW" column under "FROM," and the upper limit of the window under "TO" on Form VI PCB-1. The retention times, mean retention times and retention time windows of the surrogates are calculated and reported from the three point calibration of Aroclor 1254. If another Aroclor is substituted for 1254 in the initial calibration, that Aroclor shall be used to determine the surrogate retention time information. The Aroclor and the reason for the choice shall be noted in the SDG narrative.
- 3.13.2.8 For each Aroclor and surrogate, the Contractor shall also complete the calibration factor data on Form VI PCB-2. Prepare one form for each instrument and GC column used. Enter the calibration

factor for each chosen Aroclor peak for each standard in the initial calibration. For each Aroclor analyzed at three concentration levels, calculate and enter the mean calibration factor and the percent relative standard deviation (%RSD) under the "MEAN CF" and %RSD columns, respectively. The surrogate calibration factors, as with the surrogate retention times, are determined from Aroclor 1254 (or the substituted Aroclor). For single point calibrations, the "MEAN CF" and "%RSD" columns shall remain blank.

- 3.13.2.9 For each Aroclor and surrogate, the Contractor shall also complete the Aroclor Resolution Summary Form VI PCB-3. Prepare one form for each instrument and GC column used. Complete the header information as described in Section 3.3. Using the same assignment of first and second GC columns made for Form IV, enter the GC column number and name and identify the column length (m), internal diameter (mm), film thickness ( $\mu$ m). Enter the Lab File ID for each mid point Aroclor standard analyzed. Complete the remainder of the form using the following instructions.

- 3.13.2.9.1 Enter the retention time of the calibrated peaks for each mid point Aroclor standard (see Form VI PCB-1) in the "RT" column.
- 3.13.2.9.2 Calculate the resolution (between peaks) to the left and right of each listed peak using the definition in Exhibit D. Enter the resolution between the listed peak and the nearest peak to the left in the "Resolution Left (%)" field and the resolution between the listed peak and the nearest peak to the right in the "Resolution Right (%)" field.

3.14 GC/NPD and GC/FID Water Soluble Organics Initial Calibration (Form VI WSO/NPD and WSO/FID).

- 3.14.1 Purpose. The initial calibration involves the determination of retention times, retention time windows, calibration factors and peak resolution for each target compound and surrogate in each initial calibration standard.
- 3.14.2 Instructions. Complete one Form VI (pages 1 and 2) for **each** GC column used for each three point initial calibration. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.

Form VI WSO/NPD-1, Form VI WSO/FID-1

- 3.14.2.1 Enter the date of analysis of the standards on each form in the "Date(s) Analyzed" field. If the analysis of the standards carries over into the following day, list both the start and completion dates. Dates shall be entered as MM/DD/YY.
- 3.14.2.2 The "Level (x low)" field shall be completed by entering the concentration of the low point, mid-point, and high point calibration standards as a multiplier of the low point. Thus, the low point shall always be entered as "1.0". The concentrations of the mid-point and high point standards are specified in Exhibit D for each analytical method. The appropriate mid and high point multipliers must be determined to one decimal place and entered in the "Level (x low)" field.
- 3.14.2.3 For each target compound and surrogate, enter the actual concentration of the low point standard under the "LOW STD CONC." column on page two of Form VI.
- 3.14.2.4 Using the same assignment of first and second GC columns, enter the GC column number and identify the type of column, including the length (m), internal diameter (mm) and film thickness ( $\mu$ m) in the appropriate fields.
- 3.14.2.5 For each standard analyzed, enter the retention times of each target compound in the initial calibration, in minutes and decimal minutes, under the "RT OF STANDARDS" column on page one of Form VI. Prepare one form for each instrument and GC column used.



- 3.13.2.6 Calculate the mean retention time of each target compound and surrogate in each three point calibration, and report it in the "MEAN RT" column on page one of Form VI.
- 3.14.2.7 Calculate the retention time window for each target compound and surrogate using the specifications in Exhibit D. Enter the lower limit of the window in the "RT WINDOW" column under "FROM," and the upper limit of the window under "TO" on page one of Form VI.
- 3.14.2.8 For each target compound and surrogate, the Contractor shall also complete the calibration factor data on page two of Form VI. Enter the calibration factor of each analyte for each of the three calibration levels. Calculate and enter a mean calibration factor and the percent relative standard deviation (%RSD) under the "MEAN CF" and %RSD columns, respectively.

Form VI WSO/NPD-2, Form VI WSO/FID-2

- 3.14.2.9 Page two of Form VI shall be used to report the percent resolution between each pair of analytes, in the mid point initial calibration standard, according to the definition in Exhibit D.
- 3.14.2.10 Complete the header information as described in Section 3.3. Enter the EPA sample number for the respective standards. If simultaneous injections are **not** used, use different suffixes to identify the standards. Using the same assignment of first and second GC columns made for Form IV, enter the GC column name and identify the column length (m), internal diameter (mm) and phase thickness (µm). Enter the Date of analysis as MM/DD/YY and time of analysis in minutes and decimal minutes. Complete the remainder of the form using the following instructions.
  - 3.14.2.10.1 List each analyte in the mid point standard, in **retention time order**, including the surrogate compounds. Thus, the order of analytes in the two boxes on this form will be different due to the dissimilarity of the stationary phases of the two GC columns used. Enter the name of each target analyte in the standard as it appears on Form I. Spell out the names of the surrogates as they appear on Form VII.
  - 3.14.2.10.2 Enter the retention time of each analyte from the analysis in the "RT" column.
  - 3.14.2.10.3 Calculate the resolution between each pair of analytes. Enter the resolution between the first and second peaks on the line for the first analyte listed in the box. Enter the resolution between the second and third peaks on the line for the second analyte, and so on, until the resolutions of all possible pairs of adjacent analytes have been entered. NOTE: The last resolution field will be left blank in each table.
- 3.15 GC/MS Initial Calibration Verification Data (Form VII VOA, SV and 524.2)
  - 3.15.1 Purpose. For volatiles, semivolatiles and modified 524.2 volatiles, this form is used to report the results of the initial calibration verification standard that is analyzed immediately following the initial calibration of the GC/MS system. The results of this second source standard verify that the initial calibration standards used to calibrate the GC/MS system have not degraded or evaporated prior to analysis. Form VII is required for every initial calibration and must contain all of the required target compounds. The Contractor shall analyze the initial calibration verification standard following every initial calibration and must meet all criteria outlined in Exhibit D for the minimum RRF and maximum percent difference.
  - 3.15.2 Instructions. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.
    - 3.15.2.1 Enter the date and time of the initial calibration verification and the dates and times of the initial calibration (give inclusive dates if the initial calibration is performed over more than one

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- date). Dates shall be entered as MM/DD/YY. Times shall be reported in military time hours, minutes and decimal minutes.
- 3.15.2.2 For volatiles enter the purge method and for volatiles and modified 524.2 volatiles, enter the GC column identifier, including column length (m), internal diameter (mm) and film thickness ( $\mu\text{m}$ ).
- 3.15.2.3 Using the appropriate initial calibration (volatile, semivolatile or modified 524.2 volatile), enter the average relative response factor (RRF) for each target compound, system monitoring compound (volatiles and modified 524.2 volatiles) and surrogate (semivolatiles).
- 3.15.2.4 Calculate and report the relative response factor (RRF50 for volatiles and semivolatiles and RRF10 for modified 524.2 volatiles) from the initial calibration verification standard analysis.
- 3.15.2.5 Calculate and report the percent difference (%D) between the calculated amount and the nominal amount for each compound according to Exhibit D. Report the values in the "%D" column to one decimal place. If the %D is greater than 999.9, report as 999.9. If the %D is less than -99.9, report as -99.9.
- 3.16 GC Initial Calibration Verification Summary (Form VII WSO/NPD and WSO/FID)
- 3.16.1 Purpose. For water soluble organics (GC/NPD and GC/FID), Form VII is used to report the results of the initial calibration verification standard that is analyzed immediately following the initial calibration of the GC system. The results of this second source standard verify that the initial calibration standards used to calibrate the GC system have not degraded or evaporated prior to analysis. Form VII must accompany every initial calibration and must contain all of the required target compounds. The Contractor shall analyze the initial calibration verification standard following every initial calibration and must meet all criteria outlined in Exhibit D for retention time and maximum percent difference.
- 3.16.2 Instructions. Complete Form VII for each calibration verification standard analyzed after each initial calibration. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.
- 3.16.2.1 Enter the date and time of the initial calibration verification and the date(s) of the initial calibration (give inclusive dates if the initial calibration is performed over more than one date). Dates shall be entered as MM/DD/YY. Times shall be reported in military time in hours, minutes and decimal minutes.
- 3.16.2.2 Enter the EPA sample number and lab sample identifier of the initial calibration verification standard.
- 3.16.2.3 Using the same assignment of first and second GC columns, enter the GC column number and identify the type of column, including the length (m), internal diameter (mm) and film thickness ( $\mu\text{m}$ ) in the appropriate fields.
- 3.16.2.4 For each initial calibration verification standard analyzed, enter the retention time (in minutes and decimal minutes) of each target compound and surrogate present, under the "RT" column. Next, enter the retention time windows, established on Form VI, for each target compound and surrogate.
- 3.16.2.5 For each target compound and surrogate, enter the amount of the analyte found, to three significant figures, in the "CALC AMOUNT" column.
- 3.16.2.6 Enter the nominal amount of each target compound and surrogate in the initial calibration verification standard in the "NOM AMOUNT" column.

- 3.16.2.7 Calculate and report the percent difference (%D) between the calculated amount and the nominal amount for each compound according to Exhibit D. Report the values in the "%D" column to one decimal place. If the %D is greater than 999.9, report as 999.9. If the %D is less than -99.9, report as -99.9.
- 3.17 GC/MS Continuing Calibration Data (Form VIII VOA, SV and 524.2)
- 3.17.1 Purpose. For volatiles, semivolatiles, and modified 524.2 volatiles, this form is used to report a check of the GC/MS system calibration by the analysis of specific calibration standards. Form VIII is required for each 12-hour time period for all volatile, semivolatile and modified 524.2 volatile target compound analyses. The Contractor shall analyze calibration standards and meet all criteria outlined in Exhibit D for the minimum RRF and maximum percent difference between initial and continuing calibrations.
- 3.17.2 Instructions. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.
- 3.17.2.1 Enter the date and time of the continuing calibration and the dates and times of the initial calibration (give inclusive dates if the initial calibration is performed over more than one date). Dates shall be entered as MM/DD/YY. Times shall be reported in military time in hours, minutes and decimal minutes.
- 3.17.2.2 For volatiles enter the purge method and for both volatiles and modified 524.2 volatiles, enter the GC column identifier, including column length (m), internal diameter (mm) and film thickness (µm) as on Form V.
- 3.17.2.3 Using the appropriate initial calibration (volatile, semivolatile or modified 524.2 volatile), enter the average relative response factor (RRF) for each target compound, system monitoring compound (volatiles and modified 524.2 volatiles), and surrogate (semivolatiles).
- 3.17.2.4 Calculate and report the relative response factor from the continuing calibration standard (RRF50 for volatiles and semivolatiles and RRF10 for modified 524.2 volatiles).
- 3.17.2.5 Calculate the percent difference (%D) for all compounds. See Exhibit D for equation. Report the values in the "%D" column to one decimal place. If the %D is greater than 999.9, report as 999.9. If the %D is less than -99.9, report as -99.9.
- 3.18 GC/EC Calibration Verification Summary for Pesticide/Aroclor analysis (Form VIII PEST-1, PEST-2)
- 3.18.1 Purpose. Form VIII is used to report the results of the Performance Evaluation Mixtures (PEMs) and the mid-point concentrations of Individual Standard Mixtures A and B that, along with the PEM, bracket each 12-hour period of sample analyses. The Contractor shall submit Form VIII for each 12-hour sequence analyzed. Form VIII PEST-1 shall be completed each time the PEM is analyzed, for each GC column used. Form VIII PEST-2 shall be completed each time the Individual Standard Mixtures are analyzed, for each GC column used.
- 3.18.2 Instructions. Complete Form VIII PEST-1 and PEST-2 for each standard reported on Form IX PEST. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.
- FORM VIII PEST-1
- 3.18.2.1 Enter the date(s) of the initial calibration(s). Give inclusive dates if the initial calibration is performed over more than one day. Dates shall be entered as MM/DD/YY.
- 3.18.2.2 Using the same assignment of first and second GC columns made for Form IV, identify the GC column number and name, including the length (m), internal diameter (mm) and film thickness (µm) in the appropriate fields.

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- 3.18.2.3 On Form VIII PEST-1, enter the EPA sample number, lab sample identifier and date and time of analysis for the instrument blank that preceded the 12-hour sequence (PIBLK). For the PEM that initiated or terminated the 12-hour sequence (PEM), enter the EPA sample number, lab sample identifier, and date and time of analysis.
- 3.18.2.4 When reporting data for the PEM at the **beginning** of the initial calibration sequence, leave the "EPA Sample No.," "Lab Sample ID," "Date Analyzed," and "Time Analyzed" fields blank for the instrument blank (PIBLK), when no instrument blank is analyzed before the PEM. When reporting **all other** PEM analyses, the instrument blank fields shall be completed.
- 3.18.2.5 In the table, report the retention time for each analyte in the PEM as well as the retention time windows.
- 3.18.2.6 For each analyte in the PEM, enter the amount of the analyte found in the PEM, to three significant figures, in the "CALC AMOUNT" column.
- 3.18.2.7 Enter the nominal amount of each analyte in the PEM in the "NOM AMOUNT" column.
- 3.18.2.8 Calculate the percent difference between the calculated amount and nominal amount for each analyte according to Exhibit D. Report the values in the "%D" column to one decimal place. If the %D is greater than 999.9, report as 999.9. If the %D is less than -99.9, report as -99.9.
- 3.18.2.9 Calculate the percent breakdown for endrin and 4,4'-DDT and the combined percent breakdown in the PEM according to Exhibit D. Enter the values for the breakdown of endrin and 4,4'-DDT in their respective fields immediately under the table.

FORM VIII PEST-2

- 3.18.2.10 The upper table on Form VIII PEST-2 contains the retention time and amount data for Individual Standard Mixture A compounds. The lower table contains the data for Mixture B. Complete the form using the instructions in Sections 3.18.2.1 through 3.18.2.8 for Form VIII PEST-1.
- 3.19 GC Continuing Calibration Summary for Aroclors only and Water Soluble Organics (Form VIII PCB, WSO/NPD and WSO/FID)
- 3.19.1 Purpose. Form VIII is used to report the results of the mid-point concentration continuing calibration standards that bracket each 12-hour period of sample analysis. The Contractor shall submit the appropriate Form VIII for each 12-hour sequence analyzed, for each GC column used. If a continuing calibration standard was analyzed more frequently than every 12-hours, the Contractor shall submit the appropriate Form VIII for each continuing calibration standard analyzed, for each column used.
- 3.19.2 Instructions. Complete Form VIII for each standard reported on Form IX. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.
- 3.19.2.1 Enter the date(s) of the initial calibration(s). Give inclusive dates if the initial calibration is performed over more than one day. Dates shall be entered as MM/DD/YY.
- 3.19.2.2 On Form VIII, enter the EPA sample number, lab sample identifier and the date and time of analysis for the instrument blank that preceded the 12-hour sequence. For the continuing calibration standard that initiated or terminated the 12-hour sequence, enter the EPA sample number, lab sample identifier, and date and time of analysis. Dates shall be entered as MM/DD/YY and time shall be entered in military time in hours, minutes and decimal minutes.

- 3.19.2.3 Using the same assignment of first and second GC columns, enter the GC column number and identify the type of column, including the length (m), internal diameter (mm) and film thickness ( $\mu\text{m}$ ) in the appropriate fields.
- 3.19.2.4 For each continuing calibration standard analyzed, enter the retention time (in minutes and decimal minutes) of each target compound and surrogate present, under the "RT" column. Next, enter the retention time windows, established on Form VI, for each target compound and surrogate.
- 3.19.2.5 For each target compound and surrogate, enter the amount of the analyte found, to three significant figures, in the "CALC AMOUNT" column.
- 3.19.2.6 Enter the nominal amount of each target compound and surrogate in the continuing calibration standard in the "NOM AMOUNT" column.
- 3.19.2.7 Calculate and report the percent difference (%D) between the calculated amount and the nominal amount for each compound according to Exhibit D. Report the values in the "%D" column to one decimal place. If the %D is greater than 999.9, report as 999.9. If the %D is less than -99.9, report as -99.9.

3.20 Internal Standard Area and RT Summary (Form IX VOA, SV and 524.2)

- 3.20.1 Purpose. This form is used to summarize the peak areas and retention times of the internal standards added to all volatile, semivolatile and modified 524.2 volatile samples, including dilutions and reanalyses, blanks, matrix spikes, and matrix spike duplicates. The data are used to determine when changes in internal standard responses will adversely affect quantification of target compounds. This form shall be completed each time a continuing calibration is performed, or when samples are analyzed under the same GC/MS instrument performance check as an initial calibration.
- 3.20.2 Instructions. Complete the header information according to Section 3.3. Complete the remainder of the form using the following instructions. If samples are analyzed immediately following an initial calibration, before another instrument performance check and a continuing calibration, Form IX shall be completed on the basis of the internal standard areas of the initial calibration using the RRF50 standard for volatiles and semivolatiles, and the RRF10 standard for the modified 524.2 volatiles. Use the date and time of analysis, the lab file identifier and the EICP areas of this standard in place of those of a continuing calibration standard.
  - 3.20.2.1 Enter the date and time of analysis of the continuing calibration standard. The date shall be entered as MM/DD/YY. The time shall be reported as military time in hours, minutes and decimal minutes.
  - 3.20.2.2 For volatiles, enter the purge method. For volatiles and modified 524.2 volatiles, enter the GC column identifier, column length (m), internal diameter (mm) and film thickness ( $\mu\text{m}$ ).
  - 3.20.2.3 From the results of the analysis of the continuing calibration standard, enter the area measured for each internal standard and its retention time (in decimal minutes) under the appropriate column in the "12 HOUR STD" row.
  - 3.20.2.4 For each internal standard listed in Table 5, calculate the upper limit of the area as the area of the particular standard plus 100 percent of its area (i.e., two times the area in the "12 HOUR STD" field), and the lower limit of the area as the area of the internal standard minus 50 percent of its area (i.e., one half the area in the "12 HOUR STD" field). Report these values in the "UPPER LIMIT" and "LOWER LIMIT" rows, respectively. Calculate the upper limit of the retention time as the retention of the internal standard plus 0.50 minutes (30 seconds), and the lower limit of the retention time as the retention time in the standard minus 0.50 minutes (30 seconds).

- 3.20.2.5 For each sample, including dilutions, reanalyses, blanks, matrix spikes, and matrix spike duplicates, analyzed under a given continuing calibration, enter the EPA sample number and the area measured for each internal standard and its retention time. If the internal standard area is outside the upper or lower limits calculated in step 4, flag that area with an asterisk (\*). The asterisk shall be placed in the far righthand space of the box for each internal standard area, directly under the "#" symbol. Similarly, flag the retention time of any internal standard that is outside the limits with an asterisk.
- 3.20.2.6 Number all pages as described in Section 3.3.

**Table 5**  
**Internal Standards**

<b>Volatile and Modified 524.2 Internal Standards</b>	<b>CAS Number</b>
IS1: Fluorobenzene (FBZ)	462-06-6
IS3: Chlorobenzene-d5 (CBZ)	3114-55-4

  

<b>Semivolatile Internal Standards</b>	<b>CAS Number</b>
IS1: 1,4-Dichlorobenzene-d4 (DCB)	3855-82-1
IS2: Naphthalene-d8 (NPT)	1146-65-2
IS3: Acenaphthene-d10 (ANT)	15067-26-2
IS4: Phenanthrene-d10 (PHN)	1517-22-2
IS5: Chrysene-d12 (CRY)	1719-03-5
IS6: Perylene-d12 (PRY)	1520-96-3

- 3.21 GC Analytical Sequence (Form IX PEST, PCB, WSO/NPD and WSO/FID)
- 3.21.1 Purpose. This form is used to report the analytical sequence for each of the GC analyses. For dual column analyses, at least one form is required for each GC column used.
- 3.21.2 Instructions. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.
- 3.21.2.1 Enter the date(s) of the initial calibration. Give inclusive dates if the initial calibration is performed over more than one day. Dates shall be entered as MM/DD/YY.
- 3.21.2.2 Using the same assignment of first and second GC columns, enter the GC column number and identify the type of column, including the length (m), internal diameter (mm) and film thickness (µm) in the appropriate fields.
- 3.21.2.3 At the top of the table, report the mean retention time of the surrogate(s) from the initial calibration sequence.
- 3.21.2.4 For every analysis associated with a particular analytical sequence starting with the initial calibration, enter the EPA sample number, lab sample identifier, and date and time of analysis. Each sample analyzed as part of the sequence shall be reported on Form IX **even** if it is not associated with the SDG.

The Contractor shall use ZZZZZ as the EPA sample number to distinguish all samples that are not part of the SDG being reported.

- 3.21.2.5 Report the retention time of the surrogate(s) for each analysis in the appropriate columns. All sample analyses shall be bracketed by compliant analyses of instrument blanks and continuing calibration standards (instrument blanks and calibration verification standards are used for Pesticide/Aroclor analyses). Given the fact that the initial calibration may remain valid for some time (see Exhibit D), it is only necessary to report the data from 12-hour periods when samples, dilutions, reanalyses, matrix spike, matrix spike duplicate, blanks, or multicomponent analytes for the 72 hour confirmation requirement in an SDG were analyzed. The Contractor shall submit Form IX for the initial calibration sequence and forms that include the continuing calibration standards (or calibration verification standards for Pesticide/Aroclor analyses) that bracket **any** and **all** samples in the SDG. While the data for time periods between the initial calibration and samples in the SDG are not a routine deliverable, the data shall be available as requested (e.g., at on-site evaluations). Non-EPA samples shall be numbered ZZZZZ.
- 3.21.2.6 Flag all the retention time values which do not meet the contract requirements by entering an asterisk (\*) in the "RT" column, under the "#" symbol. If the retention time cannot be calculated due to interfering peaks, leave the "RT" column blank for that surrogate, enter an asterisk in the last column, and document the problem in the SDG Narrative.
- 3.21.2.7 If more than a single copy of Form IX is required, enter the same header information on all subsequent pages for that GC column and instrument, and number each page as described in Section 3.3.

3.22 Pesticide/Aroclor Cleanup Summary (Form X PEST-1, PEST-2)

- 3.22.1 Purpose. This form summarizes the results of the checks performed for the three cleanup procedures employed during the preparation of pesticide/Aroclor extracts for analysis. Form X PEST-1 is used to report the results of the check of the Florisil cartridges used to process all sample extracts and to associate the lot of cartridges with particular sample results so that problems with a particular cartridge lot may be tracked across all associated samples. Form X PEST-2 summarizes the results of the calibration of the Gel Permeation Chromatography (GPC) device that shall be used to process all soil sample extracts for pesticide/Aroclor analyses. Form X PEST-3 summarizes the results of all samples within an SDG requiring sulfuric acid cleanup due to the suspected presence of Aroclors. If Aroclors were determined to not be present following the initial analysis for pesticides, the sulfuric acid cleanup and Form X PEST-3 are not required.

- 3.22.2 Instructions. Complete the header information according to the instructions in Section 3.3. Note. enter the Case number and SDG number for the current data package, regardless of the original Case for which the cartridge check was performed. Complete the remainder of the form using the following instructions.

FORM X PEST-1

- 3.22.2.1 Enter the Florisil cartridge lot number.
- 3.22.2.2 Enter the date the Florisil cartridge check solution was analyzed in the "Date of Analysis" field. The date shall be entered as MM/DD/YY.
- 3.22.2.3 Identify the two GC columns, including the length (m), internal diameter (mm) and film thickness (µm), used to analyze the samples, QC samples and required blanks. Report all results from either GC column 1 or GC column 2.
- 3.22.2.4 In the first table, enter the amount of spike added and spike recovered in nanograms (ng) for each analyte.

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- 3.22.2.5 Calculate the percent recovery to the nearest whole percent, and enter the number in the "% REC" field. Flag each spike recovery outside the QC limits (shown on the form) with an asterisk (\*). The asterisk shall be placed in the last space in the "% REC" column, under the "#" symbol.
- 3.22.2.6 In the second table, complete the "EPA Sample No.," the "Lab Sample ID," and "Date Analyzed" fields for each sample and blank that were cleaned up using this lot of Florisil cartridges.
- 3.22.2.7 Number the pages as described in Section 3.3.

FORM X PEST-2

- 3.22.2.8 On Form X PEST-2, enter an identifier for the GPC column and the date of calibration in the appropriate fields.
- 3.22.2.9 Identify the GC Columns as on Form X PEST-1 for florisil. Report all results from a single column.
- 3.22.2.10 For each of the pesticide matrix spike compounds listed in the first table, enter the amount of the spike added to the GPC column and the amount recovered, in nanograms (ng).
- 3.22.2.11 Calculate the percent recovery of each analyte, and enter these values on the form, to the nearest percent. Compare the recoveries to the QC limits shown on the form, and flag all those values outside the limits with an asterisk (\*) in the "% REC" column under the "#" symbol.
- 3.22.2.12 For each sample in the data package that was subjected to GPC under this calibration, enter the EPA sample number, lab sample identifier, and the date of **both** analyses in the second table.
- 3.22.2.13 If more than one copy of Form X PEST-2 is required, number all pages as described in Section 3.3.

3.23 GC Target Compound Identification Summary (Form XI PEST-1, PEST-2, PCB, WSO/NPD and WSO/FID)

- 3.23.1 Purpose. This form summarizes the quantitations of all target compounds detected in a given sample. It reports the retention times of the compound on both columns on which it was analyzed, as well as the retention time windows of the standard for that compound on both of these columns. In addition, it is used to report the concentration determined from each GC column, and the percent difference between the two quantitative results. Note, separate forms are used for the pesticide/Arochlor method for single component analytes and multicomponent analytes.

Form XI is required for each sample (including dilutions and reanalyses), blank, matrix spike, and matrix spike duplicate in which compounds listed in Exhibit C are reported on Form I. **Do not generate a Form XI for instrument blanks.**

- 3.23.2 Instructions. Complete the header information according to the instructions in Section 3.3. Complete the remainder of the form using the following instructions.
  - 3.23.2.1 Enter the date(s) of analysis. Dates shall be entered as MM/DD/YY.
  - 3.23.2.2 In the "Matrix" field, enter SOIL for soil/sediment samples, WATER for water samples and WASTE for oily sludge and waste samples.
  - 3.23.2.3 Enter the instrument ID and the GC column identification, including the length (m), internal diameter (mm) and film thickness (µm) for each of the two columns.
  - 3.23.2.4 For each single component analyte positively identified on both the primary and confirmatory columns, enter the name of the compound in the "ANALYTE" column as it appears on Form I.



- 3.23.2.5 Enter the retention times on each column of the compounds detected in the sample next to the appropriate column designation (1 or 2).
- 3.23.2.6 Enter the retention time windows on each column from the initial calibration standard. These data shall correspond with those on Form VI and shall be entered in a similar manner. The lower value is entered under the "FROM" column, the upper value under the "TO" column.
- 3.23.2.7 Enter the concentration calculated from each GC column under the "CONCENTRATION" column. Although the units are the same as those used on Form I, do **not** enter any units on Form XI.
- 3.23.2.8 Calculate the percent difference between the concentrations entered on this form. See Exhibit D for equation, and report to a tenth of a percent in the "%D" column. If the %D is greater than 999.9, report it as 999.9.
- 3.23.2.9 The **lower** of the two concentrations is reported on Form I for each compound. The lower concentration is used because, if present, coeluting interferences are likely to increase the calculated concentration of any target compound. If the percent difference between the calculated concentrations is greater than 25.0 percent, flag the concentration on Form I, as described previously. This will alert the data user to the potential problems in quantitating this analyte.
- 3.23.2.10 If more compounds are identified in an individual sample than can be reported on one Form XI, complete as many additional copies of Form XI as necessary, duplicating all header information and numbering the pages as described in Section 3.3.
- 3.23.2.11 Report multicomponent analytes detected in samples on Form XI. Complete the header information and GC column fields as described above. For multicomponent analytes, it is necessary to report the retention time and concentration of each peak chosen for quantitation in the target analyte in a fashion similar to that for single components. The concentrations of all peaks quantitated (three are required, up to five may be used) are averaged to determine the mean concentration. Report the lower of the two **mean** concentrations on Form I. Flag this value if the mean concentrations from the two GC columns differ by more than 25 percent, as described previously.
- 3.23.2.12 If more multicomponent compounds are identified in an individual sample than can be reported on one Form XI, complete as many additional copies of Form XI as necessary, duplicating all header information and numbering the pages as described in Section 3.3.
- 3.24 Sample Log-In Sheet (Form DC-1)
- 3.24.1 Purpose. This form is used to document the receipt and inspection of sample containers and samples. One original of Form DC-1 is required for each sample shipping container (only the hardcopy form is required). If the samples in a single sample shipping container are assigned to more than one SDG, the original Form DC-1 shall be placed with the deliverables for the SDG of the lowest alpha-numeric number, and a copy of Form DC-1 shall be placed with the deliverables for the other SDGs. The copies shall be identified as "copy(ies)," and the location of the original shall be noted on the copies.
- 3.24.2 Instructions
- 3.24.2.1 Sign and date the airbill (if present).
- 3.24.2.2 Examine the shipping container and record the presence/absence of custody seals and their condition (e.g., intact, broken) in item 1 on Form DC-1.
- 3.24.2.3 Record the custody seal numbers in item 2.
- 3.24.2.4 Open the container, locate the cooler temperature indicator bottle in the sample shipping cooler, remove the cap, and insert a

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calibrated thermometer into the cooler temperature indicator bottle. Allow a minimum of 3 minutes but not greater than 5 minutes for the thermometer to equilibrate with the liquid in the bottle. Record the temperature of the cooler in item 3 on Form DC-1 and in the SDG Narrative. If the temperature of the cooler temperature indicator exceeds 10 degrees Celsius, contact the RSCC and document the contact as well as resolution of the problem on a Communication Log. Refer to Section 4.2.1.2.3 of Exhibit A for additional information.

- 3.24.2.5 Remove the enclosed sample documentation, and record the presence/absence of chain-of-custody record(s), traffic reports or packing lists, and airbills or airbill stickers in items 4-6. Specify if there is an airbill present or an airbill sticker in item 6. Record the airbill or sticker number in item 7. Complete the header information on the form, including the log-in date.
- 3.24.2.6 Remove the samples from the shipping container(s), examine the samples and the sample tags (if present), and record the condition of the sample bottles (e.g., intact, broken, leaking) and presence of absence of sample tags in items 8 and 9.
- 3.24.2.7 Review the sample shipping documents and compare the information recorded on all the documents and samples and circle the appropriate answer in item 10.
- 3.24.2.8 Record the date and time of cooler receipt at the laboratory in items 11 and 12.
- 3.24.2.9 If there are no problems observed during receipt, sign and date (include the time) Form DC-1, the chain-of-custody record, and the Traffic Report, and write the sample numbers on Form DC-1 in the "EPA Sample #" column.
- 3.24.2.10 Record the appropriate sample tags and assigned laboratory numbers, if applicable.
- 3.24.2.11 Any comments should be made in the "Remarks" column.
- 3.24.2.12 Record the fraction designation (if appropriate) and the specific area designation (e.g., refrigerator number) in the "Sample Transfer" block. Sign and date the "Sample Transfer" block.
- 3.24.2.13 Cross out unused columns and spaces.
- 3.24.2.14 If there are problems observed during receipt or an answer marked with an asterisk (e.g., "absent\*") was circled, contact the EPA and document the contact as well as resolution of the problem on a Communication Log. Following resolution, sign and date the forms and note, where appropriate, the resolution of the problem.

3.25 Document Inventory Sheet (Form DC-2)

- 3.25.1 Purpose. The Document Inventory Sheet (Form DC-2) is used to record both the inventory of Complete SDG File (CSF) documents and the number of documents in the original sample data package which is sent to the EPA Region.
- 3.25.2 Instructions
  - 3.25.2.1 Organize all EPA CSF documents as described in Exhibit B, Sections II and III. Assemble the documents in the order specified on Form DC-2 and Sections II and III, and stamp each page with a consecutive number; however, do not number Form DC-2. Inventory the CSF by reviewing the document numbers and recording page number ranges in the columns provided on Form DC-2. The Contractor shall verify and record in the "Comments" section on Form DC-2 all intentional gaps in the page numbering sequence (for example, "page numbers not used, XXXX - XXXX, XXXX - XXXX. If there are no documents for a specific document type, enter a "NA" in the empty space.

- 3.25.2.2 Certain laboratory-specific documents related to the CSF may not fit into a clearly defined category. The Contractor shall review Form DC-2 to determine if it is most appropriate to place them under categories 7, 8, 9, or 10. Category 10 should be used if there is no appropriate previous category. These types of documents should be described or listed in the blanks under each appropriate category on Form DC-2.
- 3.25.2.3 If it is necessary to insert new or inadvertently omitted documents, the Contractor shall identify the documents with unique accountable numbers and record the unique accountable numbers and the locations of the documents in the CSF in the "Other Records" section on Form DC-2.

Exhibit B -- Section 4  
Data Reporting Forms

4.0 DATA REPORTING FORMS

The data reporting forms are shown on the following pages.